QUALITY ASSURANCE PROJECT PLAN (QAPP)

PROJECT TITLE: LETTER WORK PLAN INVESTIGATIONS/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

REVISION NUMBER: 0

REVISION DATE: May 27, 2008

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QAPP REVISION SUMMARY

Revision	*Changes/Description*	*Date*
Revision 0	Initial QAPP Version	May 26, 2008
Revision 1	QAPP Update – USEPA comments	September 29, 2008
Revision 2	QAPP Update – Additional 2011 Investigations	January 3, 2011

LIST OF ACRONYMS AND SHORT FORMS

%R	Percent Recovery
CFR	Code of Federal Regulations
COC	Chain of Custody
CRA	Conestoga-Rovers & Associates
CVAA	Cold Vapor Atomic Absorption
DC-PRA	Direct Contact – Presumptive Remedy Area
DQOs	Data Quality Objectives
EDD	Electronic Data Deliverables
ESV	Ecological Screening Values
FS	Feasibility Study
FSP	Field Sample Plan
FSS	Field Support Section
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectroscopy
ICP	Inductively Coupled Plasma
IDL	Instrument Detection Limit
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MNA	Monitored Natural Attenuation
MS	Mass Spectroscopy
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NIST	National Institute of Standards and Technology
NPL	National Priority List
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness, Sensitivity
PCBs	Polychlorinated Biphenyls

LIST OF ACRONYMS AND SHORT FORMS (Cont'd)

ppb	Parts per Billion
ppm	Parts per Million
PRGs	Preliminary Remediation Goals
PRP	Potentially Responsible Party
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SDG	Sample Delivery Group
Site	South Dayton Dump and Landfill Site
SOPs	Standard Operating Procedures
SRM	Standard Reference Materials
SW-846	"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, 3rd Edition with Updates I through III, November 1986
SVOC	Semi-volatile Organic Compounds
SDDPG	South Dayton Dump Site PRP Group
TAL	Target Analyte List
TBD	To Be Determined
TCL	Target Compound List
TestAmerica	TestAmerica, Inc.
TestAmerica-EM Lab	TestAmerica P&K Laboratory, San Bruno, California
TestAmerica-NC	TestAmerica North Canton, Ohio
TestAmerica-LA	TestAmerica Los Angeles, California
TestAmerica-WS	TestAmerica West Sacramento, California
TQL	Target Quantitation Limit
USEPA	United States Environmental Protection Agency
UAO	Unilateral Administrative Order
VAS	Vertical Aquifer Sampling
VOC	Volatile Organic Compounds

K.1.0 INTRODUCTION

United States Environmental Protection Agency (USEPA) policy requires that all work performed by or on behalf of USEPA involving the collection of environmental data be implemented in accordance with a USEPA-approved Quality Assurance Project Plan (QAPP). The QAPP is a planning document that provides a "blueprint" for obtaining the type and quantity of data needed to support the intended use(s) of the data. The QAPP integrates all technical and quality aspects of a project and documents all quality assurance (QA), quality control (QC), and technical activities and procedures associated with planning, implementing, and assessing environmental data collection operations.

This QAPP has been prepared by Conestoga-Rovers & Associates (CRA) in accordance with the Region 5 Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan Based on EPA QA/R-5 (Revision 0, June 2000); EPA Requirements for Quality Assurance Project Plans (QA/R-5) (EPA/240/B-01/003, March 2001); and EPA Guidance for Quality Assurance Project Plans (QA/G-5) (EPA/600/R-98/018, February 1998). In accordance with these documents, there are four basic groups of elements that must be included in a QAPP. These four groups, the associated elements, and QAPP Sections follow:

- Group A - Project Management, Sections K.2.0 and K.3.0. The elements in this group include all aspects of project management, project objectives, and project history.
- Group B – Data Generation and Acquisition, Sections K.4.0 and K.5.0. The elements in this group include descriptions of the design and implementation of all measurement systems that will be used during the project.
- Group C – Assessment/Oversight, Section K.6.0. The elements in this group encompass the procedures used to ensure proper implementation of the QAPP.
- Group D – Data Validation and Usability, Section K.7.0. The elements in this group cover the QA activities that occur after the data collection phase of the project is completed.

The elements that comprise project management, data generation and acquisition, assessment/oversight, and data validation and usability for the groundwater and soil investigation activities to be conducted during the investigative activities as described in the Letter Work Plans (see Section K.4.1) and the Field Sampling Plan (FSP) for the South

Dayton Dump and Landfill Site located in Moraine, Ohio (Site) are documented in this QAPP.

It is the intent of the investigative activities to collect additional data to help address data gaps at the Site and aid in the completion of a Feasibility Study (FS).

The FS will include the development and evaluation of alternatives for remedial action that will meet the remedial action objectives for the Site and protect human health and the environment by eliminating, reducing or controlling risks posed through each pathway at the Site. The primary focus of this QAPP is the investigative activities in the Letter Work Plans and the FSP.

K.2.0 PROJECT ORGANIZATION

The responsibilities of management, QA personnel, field personnel, and laboratory personnel are provided in the following subsections. Additionally, any special training/certification requirements for the project are identified and an organization chart that identifies the lines of communication among the participants in the RI/FS activities is presented herein.

K.2.1 MANAGEMENT RESPONSIBILITIES

The South Dayton Dump Site PRP Group (SDDPG) has selected CRA as technical consultant responsible for implementing the FSP investigative activities at the Site. CRA's Project Manager is ultimately responsible for ensuring that the project objectives are achieved. CRA's Project Manager has selected a project team consisting of CRA's technical personnel (engineering, chemistry, and data management), CRA's QA personnel, and fixed analytical laboratories. CRA's Project Manager for the FSP investigative activities and his specific responsibilities follow:

Steve Quigley, P.E. - Project Manager – CRA

- technical representation for the SDDPG
- overview of field activities
- overview of laboratory activities
- advise on corrective actions
- prepare and review reports
- coordinate CRA's technical group
- final evidence file custodian
- approve the QAPP

The analytical laboratory's Project Manager is responsible for ensuring that the project objectives are achieved by the laboratory. The primary laboratory selected for this project is Test America, Inc. (TestAmerica). Laboratory services shall be provided by TestAmerica's North Canton laboratory (TestAmerica-NC located at 4101 Shuffel Street NW, North Canton, Ohio 44270, Telephone No. (800) 456-9396) with support from the Los Angeles laboratory (TestAmerica-LA located at 1721 South Grand Avenue, Santa

Ana, California 97205), TestAmerica's West Sacramento Laboratory (TestAmerica-WS located at 880 Riverside Parkway, West Sacramento, California 95605), and TestAmerica's EM Lab P&K Laboratory (TestAmerica-EM Lab located at 1150 Bayhill Drive, Suite 100, San Bruno, California 94066). TestAmerica's Project Managers and their specific responsibilities follow:

Denise Heckler - Laboratory Project Manager – TestAmerica-NC

Beth Riley – Laboratory Project Manager – TestAmerica-LA

Karen Dahl – Laboratory Project Manager – TestAmerica-WS

Simone Singh – Laboratory Project Manager – TestAmerica-EM Lab

- Denise Heckler will be the lead laboratory project manager and will perform and coordinate the other laboratories in the following tasks
- ensure all resources of the laboratory are available on an as-required basis
- review final analytical reports
- approve final reports prior to submission to CRA

The USEPA Region 5 Remedial Project Manager (RPM) is responsible for overview of this project. She is also responsible for submitting this QAPP and any subsequent revisions or amendments to the appropriate USEPA personnel for review and approval and for providing approval of the QAPP. Karen Cibulskis is the RPM for FSP activities at the Site.

K.2.2 QUALITY ASSURANCE RESPONSIBILITIES

Project team members with QA responsibilities include CRA's QA Officer, CRA's Field QA Officer, and TestAmerica's QA Officers. These individuals and their specific responsibilities follow:

Paul Wiseman - QA Officer - CRA

- overview and review field QA/QC
- review laboratory QA/QC
- coordinate data validation and assessment
- advise on laboratory corrective action procedures

- prepare and review QA reports
- QA/QC representation of project activities
- approve the QAPP

Jeroen Winterink – Field QA Officer - CRA

- management of field activities and field QA/QC
- field data assessment
- internal field technical system audits
- technical representation of field activities
- prepare standard operating procedures (SOPs) for field activities
- implement and document field corrective actions, if necessary
- approve the QAPP

Dorothy Leeson - Laboratory QA Officer – TestAmerica-NC

Maria Friedman – Laboratory QA Officer – TestAmerica-LA

Pam Schemmer – Laboratory QA Officer – TestAmerica-WS

Jennifer Shim – Laboratory QA Officer – TestAmerica-EM Lab

- coordinate and provide overview of laboratory systems audits
- provided overview of QA/QC documentation
- conduct detailed data review upon request
- implement and document laboratory corrective actions, if required
- provide technical representation for laboratory QA procedures
- oversee preparation of laboratory SOPs
- approve the QAPP

The USEPA Region 5 Field Support Section (FSS) Quality Assurance Reviewer is responsible for reviewing and providing final approval of the QAPP.

K.2.3 FIELD RESPONSIBILITIES

CRA will conduct all field sampling and obtain field measurements related to sampling during the investigative activities. The specific procedures for field sample collection and field measurements are presented in the Field Sampling Plan (FSP) (CRA, July 2008).

CRA's field sampling team will consist of technical staff from CRA's Cincinnati, Ohio offices. CRA's Field QA Officer will be responsible for documenting any non-conformances and subsequent corrective actions. The Field QA Officer or any field team member can identify and report non-conformances.

K.2.4 LABORATORY RESPONSIBILITIES

TestAmerica-NC will be the primary laboratory providing all laboratory deliverables and will perform all analyses of samples collected during the Site activities, except as noted. Soil, groundwater, surface water, leachate seep, and sediment samples collected will be analyzed for Target Compound List (TCL) volatile organic compounds (VOCs), TCL semi-volatile organic compounds (SVOCs), TCL pesticides, TCL polychlorinated biphenyls (PCB), TCL herbicides, and Target Analyte List (TAL) inorganics. Groundwater samples will also be analyzed for monitored natural attenuation parameters (MNA) including alkalinity, chloride, dissolved organic carbon (DOC), hardness, nitrate, nitrite, sulfate, sulfide, and dissolved gases (ethane, ethene, and methane). TestAmerica-WS will complete dioxins and furans analysis of solid samples, TestAmerica-EM Lab will analyze soil samples for asbestos. Landfill gas samples will be collected and analyzed for select VOCs by TestAmerica-LA. In addition, select samples will be collected and analyzed for waste characterization parameters, which include (Toxic Characteristics Leachate Procedure (TCLP) VOC, TCLP SVOC, TCLP pesticides, TCLP herbicide, TCLP metals, PCB, total cyanide, total sulfide, corrosivity, and ignitability).

The specific responsibilities of laboratory personnel involved in the project follow:

Ray Risden - Laboratory Operations Manager – TestAmerica-NC

Elizabeth Winger – Laboratory Operations Manager – TestAmerica-LA

Robert Hrabak – Laboratory Operations Manager – TestAmerica-WS

Kamash Ramanathan – Laboratory Operations Manager – TestAmerica-EM Lab

- coordinate laboratory analyses
- supervise in-house chain-of-custody
- schedule sample analyses
- oversee data review
- oversee preparation of analytical reports

John McFadden - Laboratory Sample Receiving Group Leader – TestAmerica-NC

Steve Gonzoles – Laboratory Sample Receiving Group Leader – TestAmerica-LA

Chen Vu – Laboratory Sample Receiving Group Leader – TestAmerica-WS

Simone Singh – Laboratory Sample Receiving Group Leader – TestAmerica-EM Lab

- the sample receiving group leader performs and coordinates other sample custodians completing the following tasks:
- receive and inspect the incoming sample containers
- record the condition of the incoming sample containers
- sign appropriate documents
- verify correctness of chain-of-custody documentation
- notify project manager of sample receipt and inspection
- assign a unique identification number and customer number, and enter each into the sample receiving log
- controlling and monitoring access/storage of samples and extracts

K.2.5 SPECIAL TRAINING/CERTIFICATION REQUIREMENTS

CRA field sampling team members are required to have received the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) safety training and annual

8-hour refresher courses required by 29 CFR Parts 1910 and 1926. On-Site subcontractor personnel involved in invasive activities (e.g., drilling, excavation) are required to have received the same training. The subcontractor is responsible for compliance of their personnel with the applicable regulations.

TestAmerica personnel training records are maintained at the laboratory. No special training or certification requirements are required for the laboratory for this project.

The surveyor used for the project will be an Ohio-licensed surveyor.

K.2.6 PROJECT ORGANIZATION

The project organization chart is presented on Figure K-2.1.

K.3.0 PROBLEM DEFINITION/BACKGROUND INFORMATION

The purposes of the investigative activities and background information for the Site are presented in the following sections.

K.3.1 SITE DESCRIPTION

The Site is located at 1901 through 2153 Dryden Road and 2225 East River Road in Moraine, Ohio. The Site is bounded to the north and west by the MCD floodway, the Great Miami River (GMR) Recreational Trail and the GMR beyond, on the east by Dryden Road and light industrial facilities beyond, to the south east with residential and commercial properties with East River Road and a residential trailer park beyond, and to the south by undeveloped land with industrial facilities beyond. The Site has been defined in the SOW as an area of approximately 80 acres, including the Valley Asphalt plant in the northern most portion of the Site, an auto salvage yard in the southeast and a gravel pit/quarry pond to the south. The central 40 acres (described as 23 acres in some documents) of the Site was referred to as the South Dayton Dump and Landfill in some reports.

More recent information including a map in MCHD files, soil boring logs, drums found at Valley Asphalt, USEPA's air photo analysis, underground storage tank closure reports, and the deposition of Horace Boesch Jr. indicate that landfilling and/or other waste disposal/handling activities occurred across most of the Site.

The Site location is shown on Figure K-3.1. A layout of the Site, including Site buildings, surface water features, and Site and parcel boundaries, is provided on Figure K-3.2.

Chemicals detected at the Site during previous investigations (see Appendix K-A) include, but are not limited to, arsenic, barium, nickel, lead, copper, PAHs, PCBs, and chlorinated solvents. Drums containing material that was leachate toxic for cadmium and lead and contained chemicals including, but not limited to, PCBs, BTEX, and TCE were found at the Site and removed in 2000. Records also indicate asbestos waste was disposed at the Site.

K.3.2 SITE HISTORY

Landfill operations continued in the central portion of the Site until the death of the landfill's operator, Mr. Alcine Grillot, in 1996. The current owners of the properties located within the Site are Valley Asphalt, Jim City Salvage, MCD, Ronald Barnett, Kathryn A. Boesch and Margaret C. Grillot. Most of the northern portion of the Site is owned by Valley Asphalt. Site History and previous investigations completed are presented in Appendix K-A.

K.3.3 CURRENT STATUS

Additional investigation activities (as described in further detail in Section K.4.1) will be undertaken to collect additional data to help address data gaps at the Site and aid in the completion of a FS.

K.3.4 PROJECT/TASK DESCRIPTION AND SCHEDULE

Investigative activities for the Site include the implementation of the FSP.

A summary of the sampling and analysis program associated with the 2008 investigative activities is provided in Table K.3.1. Table K.3.2, Table K.3.3, Table K.3.4, and Table K.3.5 provide the parameter lists and associated targeted quantitation limits for samples collected during the investigative activities.

K.4.0 DATA QUALITY OBJECTIVES

Data quality objectives (DQOs) are qualitative and quantitative statements derived from the outputs of each step of the DQO process. The DQO process is a series of planning steps based on the scientific method that is designed to ensure that the type, quantity, and quality of environmental data used in decision making are appropriate for the intended application.

There are seven steps in the DQO process which include:

- Step 1: State the Problem - The contamination problem that will require new environmental data is summarized, and the resources available to resolve the problem are identified. The budget and schedule are examined and the key decision makers are identified and members of the Planning Team are selected. The decision-makers include SDDPG, the USEPA Region 5, Ohio EPA and CRA as technical consultant responsible for implementing investigative activities at the Site.
- Step 2: Identify the Decision - The decisions that require new environmental data to address the contamination problem are identified.
- Step 3: Identify the Inputs to the Decision - The information needed to support the decision is identified, and which inputs require new environmental measures are identified.
- Step 4: Define the Study Boundaries - The spatial and temporal aspects of the environmental media that the data must represent are identified.
- Step 5: Develop a Decision Rule - A logical "if…then" statement that defines the conditions that would cause the decision maker to choose among alternative actions is developed.
- Step 6: Specify Limits on Decision Errors - Acceptable limits on decision error, which are used to establish goals for limiting uncertainty in the data, are specified.
- Step 7: Optimize the Design for Obtaining Data - The most resource-efficient sampling and analysis design for generating data that are expected to satisfy the DQOs is specified.

The data collected from the Letter Work Plan Investigations will be used to assist in developing and evaluating alternatives for remedial action that will meet the remedial action objectives for the Site.

Sampling data from previous investigations and data from the proposed investigative activities need to be assessed against a set of screening criteria to determine concentrations of significance. In general, establish target quantitation limits (TQLs) for the investigative activities should be as low or lower than the screening criteria. As an initial screen to evaluate data, and potential risks to human health, sampling data for groundwater will be compared to the Preliminary Remediation Goals (PRGs) established by USEPA Region 9 (USEPA October 2004). The PRG criteria are provided in Table K.4.1. To assess potential risks to ecological receptors when groundwater discharges to nearby surface water, groundwater concentrations will be compared to surface water criteria and screening values from the following sources. Surface water / Groundwater quality criteria for protection of aquatic life from Ohio will initially be chosen. If no Ohio criterion is available, then national water quality criteria (USEPA, 1999b) for freshwater will be used. If no national water quality criteria are available, then Ecological Screening Values (ESVs) from USEPA Region V or water quality criteria from the state of Michigan will be used as ESVs. In all cases, more conservative chronic aquatic life criteria will be used as ESVs. Based on sampling results from a nearby Site, hardness related criteria were estimated at 100 mg/L hardness for surface water. The ESVs for screening groundwater results are presented in Table K.4.2.

The TQLs are presented in Tables K.3.2, K.3.3, and K.3.4. In certain cases, the groundwater PRGs are lower than the targeted quantitation limits identified in Table K.3.2. Similarly, some of the ESVs are also below the TQLs. In these cases, the estimated concentrations reported below the TQL to the laboratory's method detection limit will be provided for these analyses. However, the PRGs for several compounds still will not be achievable using this approach. This is a limitation of the standard analytical methods.

The spatial boundaries of the delineation actives are the physical boundaries of the Site, as described in the draft RI/FS Work Plan.

The limits on decision errors primarily relate to the level of accuracy of the environmental measurements as they are compared to the screening criteria provided on Table K.4.1. Error can be introduced during the sample collection, handling, preparation, analysis, data reduction, or data handling phases of the data collection

process. The acceptable levels of measurement performance criteria are provided in Section K.4.2 for field and laboratory precision, accuracy, compatibility, and

completeness. Data will be evaluated through the verification and validation process to ensure that suitable level of precision, accuracy, compatibility, and completeness is achieved for the measurement data. Professional judgment will be used to determine practical versus statistical significance of test results.

The sampling strategy includes a degree of flexibility, such as the addition of monitoring well locations to the current network, so that the sample design can be optimized to ensure sufficient data are collected to evaluate the remedial alternatives.

K.4.1 SAMPLING ACTIVITIES

The proposed sampling activities include the following:

Section K.4.1.1 Soil, Test Trenches, and Test Pits;

Section K.4.1.2 Groundwater;

Section K.4.1.3 Surface Water;

Section K.4.1.4 Sediment;

Section K.4.1.5 Landfill Gas/Soil Vapor; and

Section K.4.1.6 Leachate Seep Investigation.

Section K.4.1.7 Sub-slab soil vapor and indoor air sampling activities

Sample locations are detailed in the corresponding letter work plans, which have been previously submitted and are provided as Appendices to the QAPP as detailed below:

- Test Pit/Test Trench Investigation Letter Work Plan (CRA, May 9, 2008) provided as Appendix K-B
- Groundwater Letter Work Plan (CRA, May 7, 2008) provided as Appendix K-C
- Landfill Gas/Soil Vapor Investigation Letter Work Plan (CRA, July 21, 2008) provided as Appendix K-D
- Leachate Seep Investigation Letter Work Plan (CRA, May 6, 2008) provided as Appendix K-E
- Vapor Intrusion Investigation Work Plan (CRA, December 17, 2010) provided as Appendix K-L.

K.4.1.1 SOIL, TEST TRENCHES AND TEST PITS

The objectives of the test pit and test trench excavation and sampling are as follows:

- collect data to assist in identifying the nature and delineating the extent of various types of landfilled materials above the water table;
- collect data to assist in characterizing landfill materials above the water table;
- collect data to assist in characterizing leachate from unsaturated landfilled material;
- assess areas of the Site previously identified as specific areas of concern [i.e., Valley Asphalt drum removal area, Valley Asphalt former underground storage tank (UST) area (a.k.a. Dayton Recycling), Custom Delivery UST area, Lot 4423, etc.]; and
- identify Site areas, which may require further investigation (for example leachate sampling and analysis, groundwater quality investigation, or other delineation work).

All soil samples will be described based on visual observations by an on-Site geologist using the Unified Soil Classification System (USCS) and will be screened using a PID.

Detailed sampling requirements are presented in the Letter Work Plans referenced above and the FSP. Selected samples will be submitted to a laboratory for analysis. Soil sample chemical analyses are provided in the Letter Work Plans listed above and summarized in Table K.3.1.

Test pits and test trenches are proposed in locations where the PRP Group would like to collect additional information about the depth and nature of the fill material above the water table. The information will be used to verify the limits of fill and to assist in characterizing the nature of the landfilled materials present in the areas investigated.

Six test pits will be excavated in the central portion of the Site. Twenty-three test trenches will be excavated throughout the Site.

The locations of the test pits and test trenches will be finalized based on the results of the geophysical investigation (the USEPA may be asked to approve moving, relocating, or adding test pit and test trench locations based on field observations, geophysical

investigation results, etc.). The nature and depth of fill material above the water table will be visually identified and recorded. Test trenching will focus on the perimeter of the PRP Group's preliminary direct contact presumptive remedy area, which was defined in the Remedial Investigation/Feasibility Study (RI/FS) Statement of Work (SOW) and the area immediately beyond the perimeter. In addition, the test trenching will assist in identifying and characterizing fill material at locations along the western embankment of the Site. Excavations will be completed to the depth of the water table, where possible (as limited by the ability of the excavator to reach the depth of the water table, the stability of the walls of the excavation, and/or the presence of obstructions). If an obstruction is encountered during the excavation of a test trench, the location of the trench will be adjusted to avoid the obstruction. If excavation to the water table is not possible due to the depth of the water table or the stability of the fill material, the PRP Group will consider the need for additional investigation at the location in question during future investigation work. The potential impacts from saturated fill materials will be assessed as part of the groundwater investigation proposed for the Site (under separate cover). The utility of this information to the FS is discussed above.

Surface soil samples will be collected during the leachate seep investigation if seeps are identified and an adequate volume of leachate for analysis cannot be collected and subsurface soil samples will be collected from test pit/ test trenches. Composite waste/fill samples will also be analyzed from the test pits and test trenches.

The following protocol will be used to determine the number of samples to be submitted for laboratory analysis. Specific samples to be submitted for laboratory chemical analysis will be selected by the CRA field representative and reviewed with the USEPA's Site representative(s) on a daily basis. Depending on the nature of materials encountered in an individual test pit or trench, the number of samples for each medium may vary. For example, if no drums or only minimal amounts of drum remnants are observed in a test pit, samples of drum contents would not be collected. In addition, the number of samples submitted for laboratory chemical analysis may increase or decrease depending on headspace results, field observations, the spatial distribution and types of existing data, and the number and types of samples collected.

The intent of the test pit and test trench investigation is to identify locations that exhibit similar characteristics (i.e., visual, physical, and, to the extent the materials are analyzed, chemical composition). Test pits may be grouped together based on similar field

observations. Where grouping occurs, CRA will select samples from the entire grouping for chemical analysis. Sample selection will be performed such that fill types from multiple different locations are selected. The parameters and associated analytical methods are specified in Tables K.3.1, and K.5.3. The composite samples will be analyzed using TCLP methods for the parameters listed in Table K.3.1.

K.4.1.2 GROUNDWATER

The objectives for the phases of work associated with groundwater are as follows:

- define subsurface stratigraphy, including identifying till-rich zone(s) and sand and gravel aquifer zone(s) at locations beneath the Site to a depth of 100 feet below ground surface using Rotosonic drilling;
- collect data to assist in characterizing groundwater impact;
- recognizing that there may be seasonal or event-related differences in groundwater elevation, flow conditions and contaminant concentrations, and that there may be more than one contaminant flow path and more than one source of groundwater contamination at the Site, attempt to: i) determine the appropriate screened interval(s) for shallow monitoring wells at Vertical Aquifer Sampling (VAS) locations through VAS data; ii) compare the screened intervals identified through VAS to the screened intervals and screen lengths in the existing wells; and iii) determine, based on these results and all existing data for the Site, if the screened intervals and screen length of the existing wells represent a zone of chemical impact in the shallow aquifer that is worthwhile to continue to monitor or not;
- characterize groundwater chemistry at Site monitoring wells through groundwater sampling and laboratory analysis; and
- collect groundwater and surface water elevation measurements over time to identify horizontal hydraulic gradients, flow directions, and, if nested wells are proposed in Phase 2, vertical hydraulic gradients.

Phase 2 will consist of three main work tasks – monitoring well installation, groundwater sampling, and continuous hydraulic monitoring.

The specific rationale for well locations will be developed after the completion of vertical aquifer sampling (VAS) at the Site. VAS and groundwater sampling are detailed in the Groundwater Letter Work Plan, which is provided as Appendix K-C. Groundwater samples will be collected and analyzed for all or a subset of the parameters listed in Table K.3.1, as appropriate, in accordance with the sampling procedures in the FSP.

Filtering is an important process to remove suspended particulate that affect sample results. Filtration of groundwater samples is generally limited to metals and DOC analysis.

Filtering can be completed in the field using in-line filters or a vacuum filter kit. Filtering of samples can also be completed by the laboratory, in which case the samples must not be preserved and must be at the laboratory within at least 24 hours of sample collection.

K.4.1.3 SURFACE WATER

The objective of the surface water sampling (if required) is as follows:

- verify groundwater/surface water interactions, groundwater migration, and human health and ecological risks associated with exposure to Site surface water.

If surface water and sediment samples are collected at the same location, the surface water samples will be collected first. If collected, surface water samples will be collected and analyzed for all or a subset of the parameters listed in Table K.3.1, as appropriate, in accordance with the sampling procedures in the FSP.

K.4.1.4 SEDIMENT

The objective of the sediment sampling (if required) is as follows:

- characterize sediments and determine the nature and extent of sediment migration and contaminant adsorption.

If collected, sediment samples will be analyzed for all or a subset of the parameters listed in Table K.3.1, as appropriate, in accordance with the sampling procedures in the FSP.

K.4.1.5 LANDFILL GAS/SOIL VAPOR

The objectives of the landfill gas/soil vapor sampling are as follows:

- assess the presence of LFG and soil vapor concentrations at locations within the Site, including properties along Dryden Road;
- obtain current data in locations where historic information indicated potential landfill gas generation concerns;
- develop information to assist in calculating future landfill gas generation rates for the FS[1]; and
- develop information to assist in evaluating the need for and type of landfill gas control at the Site for the FS.

Soil gas probes will be installed in the vicinity of the Site in accordance with the Landfill Gas/Soil Vapor Investigation Letter Work Plan provided in Appendix K-D. Four of the 20 gas probes are located within the limits of the Preliminary Direct Contact Risk – Presumptive Remedy Area (DC-PRA) and will provide information with respect to LFG/soil vapor generation within known municipal waste landfill areas. The scope and location of the gas probes has also taken the closest receptors into consideration. A total of 14 gas probe locations are proposed for installation along Dryden Road. Twelve of the sixteen gas probes are located on commercial properties within 50 feet of occupied structures on Dryden Road. These gas probes will provide data with respect to the risk to occupants of adjacent buildings from LFG and soil vapor migration from the Site. The soil gas probe installation procedures are presented in Section J.2.6 of the FSP. Further details regarding the soil gas probe sampling protocol are presented in Appendix J-J of the FSP.

1 The requirements for the explosive gas monitoring plan specified in OAC 3745-27-12 will be assessed once it is known if there is explosive gas issues associated with this landfill that has been closed for more than 30 years.

If collected, soil gas samples will be analyzed for all or a subset of the parameters listed in Table K.3.1, as appropriate. As discussed in Appendix K-D, field measurements of gas pressure, methane (v/v), combustible gases (lower explosive limit, LEL), and oxygen (v/v) will be made.

K.4.1.6 LEACHATE SEEP INVESTIGATION

The objectives of the leachate seep investigation are as follows:

- complete a seep inspection to identify the location, extent, and characteristics of seeps observed along Site embankments and in other on-Site and near-Site areas;
- characterize seeps observed along Site embankments and in other areas; and
- identify any area(s) that may require further investigation.

In accordance with the Leachate Seep Investigation Letter Work Plan provided in Appendix K-E, this assessment will consist of a visual inspection of the entire embankment surface, nearby areas, and low lying areas with an objective to document any evidence of groundwater or leachate discharge from any portion of the bank and other nearby or low-lying areas. Specific items to be investigated include identifying erosion rills, areas of surface staining and/or stressed vegetation, and wet or saturated areas resulting from seeping liquid.

Should leachate seeps, surface staining, stressed vegetation, or other evidence of a leachate seep be identified in any of the embankments or in other areas CRA will collect leachate and/or soil samples (as detailed below) at the identified location. If no active seep is observed but indirect evidence of a seep is observed (erosion rills, stressed vegetation, etc.), then CRA will collect a surface soil sample from the area where the observation was made.

Leachate and leachate seep soil samples will be collected and analyzed for all or a subset of the parameters listed in Table K.3.1, as appropriate, in accordance with sampling procedures in the FSP.

K.4.1.7 SUB-SLAB SOIL VAPOR AND INDOOR AIR SAMPLING ACTIVITIES

The objectives of the sub-slab soil vapor sampling are as follows:

- Determine whether contaminant concentrations pose more than a 1×10^{-4} cancer risk or a hazard index (HI) greater than 1.0 through the vapor intrusion (VI) pathway to current or potential future receptors
- Determine whether concentrations of combustible gases within a structure exceed 10 percent of the Lower Explosive Limit (LEL) for methane)
- Identify buildings where indoor air sampling is required based on the sub-slab sample results

Sub-slab soil vapor probes will be installed in accordance with the Vapor Intrusion Investigation Work Plan, dated December 17, 2010. Sub-slab soil vapor probes will be installed beneath the following existing on-Site structures:

A. Structures On Site West of Dryden Road:
3 building structures on Lot 5054
3 building structures on Lot 5171
2 building structures on Lot 5172
1 building structure on Lot 5173
1 building structure on Lot 5174
1 building structure on Lot 5175, and

B. Structures On Site or Adjacent to Site Along East River Road:
4 building structures on Lot 4610 (Barnett; on-Site)
2 building structures on Lot 3207
1 residence on Lot 3253; and
1 building structure on Lot 3254.

Prior to conducting the sub-slab soil vapor sampling, CRA will visually inspect the Lots in question and document the number and type of buildings present on each Lot in order to ensure that all buildings that are or may be occupied are included in the sampling program.

Prior to installing the sub-slab soil vapor probes, a survey will be conducted of each building, to identify potential preferential pathways for vapor migration under the building. Details of the building survey are included in the Vapor Intrusion

Investigation Work Plan. If any structure on or adjacent to the Site that is or may be occupied has no slab (e.g., dirt or gravel floor), indoor air samples will be collected. For any location where an indoor air sample is collected, CRA will also install a soil vapor probe screened between 3 and 5 feet below ground surface in accordance with CRA's SOP [Appendix J-F-11 of the Field Sampling Plan (FSP)] in order to attempt to correlate indoor air concentrations to concentrations of contaminants in soil vapor near the structure. The soil vapor probes will be installed immediately adjacent to the side of the building closest to the most likely source of any soil vapor impacts. In addition, where indoor air samples are collected, CRA will also collect ambient air samples immediately adjacent to the structure as per CRA's SOP.

CRA's standard operating procedure (SOP) for installing sub-slab probes and collecting sub-slab vapor samples are in Attachment A to the Vapor Intrusion Investigation Letter Work Plan (addendum to the FSP). CRA's SOP for indoor air sampling is in Attachment B to the Vapor Intrusion Letter Work Plan (addendum to the FSP).

If collected, sub-slab soil gas samples will be analyzed for benzene, toluene, ethylbenzene, and xylenes (BTEX), along with chlorinated VOCs including perchloroethylene (PCE), trichloroethylene (TCE), cis/trans-1,2-dichloroethylene (1,2-DCE), 1,1-dichloroethylene (1,1-DCE), and VC in accordance with the USEPA Toxic Organics-15 (TO-15) parameter list.

K.4.2 MEASUREMENT PERFORMANCE CRITERIA

The measurement performance criteria for precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) are provided in the following subsections.

K.4.2.1 PRECISION

Precision is a measure of the degree to which two or more measurements of the same characteristic (i.e., analyte, parameter, etc.) under the same or similar conditions are in agreement.

K.4.2.1.1 FIELD PRECISION CRITERIA

Precision of the field sample collection procedures will be assessed by the data from analysis of field duplicate samples. Relative percent differences (RPDs) will be calculated for detected analytes from field duplicate sample sets. Field duplicate samples will be collected at a minimum frequency of one per 10 investigative samples. RPDs of 50 percent for water sample field duplicates will be used as advisory limits. Professional judgment will be used for any data qualification.

Field precision for measurements obtained during well stabilization monitoring will be assessed through the collection and measurement of duplicate samples or calibration solutions at a frequency of one per 10 or fewer field measurements. The precision acceptance criteria for field measurements obtained during the field activities are presented in the SOPs in Appendix K-F.

K.4.2.1.2 LABORATORY PRECISION CRITERIA

Laboratory precision will be assessed through the calculation of RPDs for replicate/duplicate sample analyses. In general, these will be matrix spike/matrix spike duplicate (MS/MSD) for water and soil samples while laboratory control sample/laboratory control duplicate (LCS/LCD) are used for air samples. The equation to be used to determine precision is presented in Section K.7.2.2 of this QAPP. Current laboratory precision control limits for the analyses are presented in Table K.4.3 and the TestAmerica Reference Data Summary provided in Appendix K-G.

K.4.2.2 ACCURACY

Accuracy is the extent of agreement between an observed value (i.e., sample result) and the accepted or true value for the parameter being measured.

K.4.2.2.1 FIELD ACCURACY CRITERIA

The criteria for accuracy of the field sample collection procedures will be to ensure that samples are not affected by sources external to the sample, such as sample

contamination by ambient conditions or inadequate equipment decontamination procedures. Field sampling accuracy will be assessed by the data from field and trip blank samples.

Field blank samples will be collected at a frequency of one per 10 sampling equipment decontamination procedures or a least once per day of sampling equipment cleanings, whichever is more frequent. Field blank samples will be collected by routing laboratory-provided deionized water through decontaminated sampling equipment. Field blank samples will be analyzed to check procedural contamination and/or ambient conditions and/or sample container contamination at the Site that may cause sample contamination. Field blank samples will not be collected for the samples collected using pre-cleaned or clean, disposable sampling equipment.

Field blank samples should not contain target analytes. The field blank sample data will be evaluated using the procedures specified in Section K.7.2.3 of the QAPP. Accuracy will be ensured by adhering to all sample-handling procedures, sample preservation requirements, and holding time periods.

Accuracy of field measurements obtained during groundwater monitoring will be assessed by analyzing calibration check samples. Accuracy acceptance criteria for field measurements obtained during the field activities are presented in the SOPs in Appendix K-F.

K.4.2.2.2 LABORATORY ACCURACY CRITERIA

Laboratory accuracy will be assessed by determining percent recoveries from the analysis of laboratory control samples (LCSs) or standard reference materials (SRMs). Accuracy relative to the sample matrix will be assessed by determining percent recoveries from the analysis of MS/MSD samples. MS/MSD samples will be collected/designated for the analyses at a minimum frequency of one per 20 or fewer samples. The equation to be used to determine accuracy for this project is presented in Section K.7.2.3 of this QAPP. Current laboratory accuracy control limits are presented in Table K.4.3 and in the TestAmerica Reference Data Summary provided in Appendix K-G.

The accuracy of the organics analyses also will be monitored through the analysis of surrogate compounds. Surrogate compounds are added to each sample, standard, blank, and QC sample prior to sample preparation and analysis. Surrogate compounds are not expected to be found occurring naturally in the samples, but behave analytically similar to the compounds of interest. Consequently, surrogate compound percent recoveries will provide information on the effect that the sample matrix exhibits on the accuracy of the analyses. Table K.4.4 and the TestAmerica Reference Data Summary provided in Appendix K-G provides current laboratory surrogate compound percent recovery control limits for the organic analyses.

K.4.2.3 REPRESENTATIVENESS

Representativeness is a qualitative term that describes the extent to which a sampling design adequately reflects the environmental condition of a site. Representativeness also reflects the ability of the sample team to collect samples and laboratory personnel to analyze those samples in such a manner that the data generated accurately and precisely reflect the conditions at a site.

K.4.2.3.1 FIELD REPRESENTATIVENESS CRITERIA

Representativeness is dependent upon the proper design of the sampling program. The representativeness criteria for field sampling will be to ensure that the sampling grids are properly established at the site, that the correct monitoring wells are sampled, and that the sampling procedures in the Appendix J-J of the FSP are followed. The sampling programs were designed to provide data representative of Site conditions. During development of these programs, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes, and constraints inherent to the Superfund program.

K.4.2.3.2 LABORATORY REPRESENTATIVENESS CRITERIA

The representativeness criteria for laboratory data will be to ensure that the proper analytical procedures are used for sample preparation (e.g., homogenizing the sample prior to subsampling), sample analysis, and that sample holding times are met.

Additionally, the accuracy and precision of the laboratory data affect representativeness. The laboratory representativeness criteria will include achieving the accuracy and precision criteria for the sample analyses.

K.4.2.4 COMPARABILITY

Comparability is an expression of the confidence with which one data set can be compared with another.

K.4.2.4.1 FIELD COMPARABILITY CRITERIA

The criteria for field comparability will be to ensure and document that the sampling networks designed for the FSP activities are properly implemented and the sampling procedures in the Appendix J-J of the FSP are followed for the duration of the sampling programs.

K.4.2.4.2 LABORATORY COMPARABILITY CRITERIA

The criteria for laboratory data comparability will be to ensure that the analytical methods used for the investigative sampling and analysis events that are comparable to the methods used for previous sampling events. The analytical methods identified in Section K.5.3.2 of this QAPP are comparable to the methods used to generate data for previous investigations.

K.4.2.5 COMPLETENESS

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data that were expected to be obtained under normal conditions.

K.4.2.5.1 FIELD COMPLETENESS CRITERIA

The criteria for field completeness will be that a minimum of 90 percent of the field-measured data are valid. The procedure for determining field data validity is provided in Section K.5.9.2 of this QAPP. The equation for calculating completeness is presented in Section K.7.2.5 of this QAPP.

K.4.2.5.2 LABORATORY COMPLETENESS CRITERIA

The criteria for laboratory completeness will be that a minimum of 90 percent of the laboratory data will be determined to be valid (usable) for the intended purpose. The procedure for determining laboratory data validity is provided in Section K.5.9.2 of this QAPP. The equation for calculating completeness is presented in Section K.7.2.5 of this QAPP.

K.4.2.6 SENSITIVITY

Sensitivity is the ability of a method or instrument to detect a parameter to be measured at a level of interest.

K.4.2.6.1 FIELD SENSITIVITY CRITERIA

The sensitivity of the field flow-through cell multi-meters selected to monitor well stabilization for this project will be measured by analyzing calibration check solutions, where appropriate, that equate to the lower end of the expected concentration range.

K.4.2.6.2 LABORATORY SENSITIVITY CRITERIA

The sensitivity requirements for the laboratory analyses are defined by the targeted quantitation limits (TQL) and method detection limits (MDL) which are provided in Table K.3.2, Table K.3.3, Table K.3.4, Table K.3.5, and Appendix K-G. The evaluation criteria for this sampling program are the USEPA Region 9 Preliminary Remediation Goals (PRGs) provided in Table K.4.1. In certain cases, the groundwater PRGs are lower

than the targeted quantitation limits identified in Table K.3.2. In these cases the estimated concentrations reported below the TQL down to the laboratory's method detection limit will provided for these analyses. However, the PRGs for several compounds still will not be achievable using this approach.

K.4.3 SPECIAL TRAINING/CERTIFICATION REQUIREMENTS

Special training/certification requirements for this project were provided in Section K.2.5.

K.4.4 DOCUMENTATION AND RECORDS

The documents, records, and reports generated during the investigative activities are identified in the following subsections.

K.4.4.1 FIELD AND LABORATORY RECORDS

Documents and records generated during the project include sample collection records, QC sample records, field measurement records, laboratory records, and data handling records. A brief description of these documents and records are provided below. Detailed information on these records is provided in subsequent sections of this QAPP.

Sample collection records that will be used during the sampling activities include field logbooks, stratigraphic logs, sample labels, chain-of-custody records, and shipping papers.

QC sample records that will be used during the project to document the generation of QC samples include field logbooks for recording field duplicate samples and MS/MSD samples. TestAmerica will maintain appropriate documentation of trip blank sample preparation, quality records for deionized water sent for field blank samples, and sample integrity information. Records of sample preservation will be maintained in field logbooks and by TestAmerica.

Field measurements will be recorded in bound logbooks. Calibration data, where applicable, will also be recorded in these logbooks.

Laboratory records that will be maintained for the project include sample receipt documentation, field and laboratory chain-of-custody documentation, sample container cleanliness certifications, reagent and standard reference material certifications, sample preparation records, sample analysis records (e.g., run logs), instrument/raw data, QC data, calibration data, corrective action reports, and final reports.

Data handling records that will be maintained include verification of computer programs used to manipulate or reduce raw data into final results and data validation reports. TestAmerica will maintain documentation of data verification and reduction procedures as necessary for the analyses used during the investigative activities. CRA will maintain checklists, notes, and reports generated during the external data validation process.

K.4.4.2 DATA REPORTING FORMAT

Field data will be recorded in bound logbooks or on standard forms (stratigraphic logs). The details for recording field data are provided in Section K.5.2.2.1 of this QAPP. Field data will be primarily generated from direct-reading meters or consist of field readings (e.g., depth to water measurements) or observations. These data will be tabulated and included in project reports or submittals.

Laboratory reports for the investigative activities include two levels of data deliverables depending on the data validation level required. These two report data deliverables are described below:

QC Summary Report - Reduced Data Validation

i) Title Page:

- project name and number;
- laboratory project or lot number;
- signature of the Laboratory QA Officer or his designee; and
- date issued.

i) Table of Contents - laboratory report contents

ii) Case Narrative:

- number of samples and respective matrices;
- laboratory analysis performed;
- any deviations from intended analytical strategy;
- definition of data qualifiers used;
- QC procedures utilized and references to the acceptance criteria;
- condition of samples "as received";
- discussion of whether or not sample holding times were met;
- discussion of technical problems or other observations which may have created analytical difficulties; and
- discussion of laboratory QC checks which failed to meet project criteria.

i) Analytical Methods Summary - methods of sample preparation and analyses for samples.

ii) Analytical Sample Summary - cross-reference table of laboratory sample to project sample identification numbers.

iii) Shipping and Receiving Documents:

- sample container documentation; and
- sample reception information and original chain of custody record.

i) Chemistry Data Package by Analysis:

- Sample Results:
  - CRA and laboratory sample identification numbers;
  - dates and times of sample collection, reception, preparation, and/or analysis;
  - sample specific quantitation (report) limits (RL), reporting MDL and estimated values between the RL and MDL;
  - methods of sample preparation and analyses for samples; and
  - dilution factors.
- QC Summary Data with Current Control Limits:
  - method blank results;

- surrogate recoveries (organics);
- matrix spike and matrix spike duplicate recoveries;
- laboratory control samples (laboratory control duplicates): and
- matrix duplicate relative percent differences.

Laboratory QC summary data deliverables will be provided to CRA within 14 days from the date of sample log-in for analysis at the laboratory.

Expanded Report – Full Data Validation

These report deliverables include those in the QC Summary reports identified above with the following additional items.

- Chemistry Data Package by Analysis
  - QC Summary Data with Current Control Limits:
    - GC/MS tuning results (organic);
    - Internal standards;
    - Interference check standards (inorganics);
    - Serial dilutions.
  - Standard Data:
    - initial calibration data, initial calibration checks, continuing calibration verification/check standards;
    - initial and continuing calibration blanks; and
    - raw data for calibration data (data chromatograms, parameter specific quantitation reports, mass spectra and instrument printouts.
  - Raw Data:
    - Dated chromatograms, parameter specific quantitation reports, mass spectra and instrument printouts of all samples and QC samples;
    - Instrument run logs;
    - Sample preparation records; and
    - Instrument conditions.

Laboratory expanded data deliverables will be provided to CRA within 21 days from the date of sample log-in for analysis at the laboratory.

K.4.4.3 DATA ARCHIVING AND RETRIEVAL

A 10-year maintenance period is required following completion of the remedial action. All records will be maintained for a period of 6 years following the 10-year maintenance period. USEPA is to be notified 90 days prior to disposal or destruction of records.

K.5.0 DATA GENERATION AND ACQUISITION

The design and implementation of the measurement systems that will be used during the investigative activities, including sampling procedures, analytical procedures, and data handling and documentation are detailed in the following subsections.

K.5.1 SAMPLING PROCESS DESIGN

The rationale for the investigative activities is provided in the FSP and the Letter Work Plans. The sampling program was developed based on the Site inspections conducted by CRA, review of existing data, and refined through planning meetings.

K.5.1.1 SAMPLING METHODS

Sampling methods for the collection of soil, groundwater, surface water and sediment samples are provided in Appendix J-J of the FSP.

K.5.1.2 FIELD EQUIPMENT AND SAMPLE CONTAINER CLEANING PROCEDURES

Equipment cleaning/decontamination procedures are provided in Appendix J-J of the FSP. Sample containers will be provided by TestAmerica. TestAmerica's vendor for sample containers is ESS of Jackson, Michigan. All containers will be pre-cleaned in accordance with the USEPA guidance document entitled "Specifications and Guidance for Contaminant-Free Sample Containers", EPA 540/R-93/051. Certificates of analysis for each lot of containers will be maintained by TestAmerica.

K.5.1.3 FIELD EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION REQUIREMENTS

Field equipment will be inspected and tested prior to being shipped to the field. Maintenance logs for all field equipment are kept in CRA's field equipment files at the CRA office from which the equipment was shipped. Prior to use in the field, the equipment is checked again, generally during field calibration, and the performance

information is recorded in the field logbook. All equipment shipped back from the field is inspected and tested upon return. Any required maintenance is performed and documented prior to the equipment being returned to service. Example field equipment maintenance, testing, and inspection forms are provided in Appendix K-H.

Critical spare parts for field equipment and replacement field equipment are available at each CRA office and can be shipped for overnight delivery, picked up at the CRA office, or delivered to the field when the need is identified. Alternately, field equipment vendors (e.g., Hazco) can provide replacement equipment if needed. The replacement equipment can be shipped for overnight delivery as necessary. A list of critical spare parts for field equipment is provided in Table K.5.1.

K.5.1.4 INSPECTION AND ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND SAMPLE CONTAINERS

The field supplies for the investigative activities consist of calibration standard solutions for field instrument calibration and calibration checks, detergent (Alconox) for equipment cleaning, distilled water for sample collection equipment rinsing, deionized water for final sample collection equipment rinsing, chemical preservatives for pH adjustment of the appropriate aliquots of aqueous samples, and sample containers to collect the water and soil samples.

CRA's Field QA Officer is ultimately responsible for ensuring that the field calibration standards for the project are acceptable. The calibration standards will be checked prior to being sent to the field to ensure that they have not expired or otherwise degraded. New calibration standards will be purchased if existing standards are found to be expired or degraded. Alconox, which is a standard laboratory-grade detergent, also is obtained from Cole Parmer. Distilled water will be purchased as needed from a variety of vendors.

Deionized water, purge-and-trap grade water, chemical preservatives, and sample containers will be provided by TestAmerica. TestAmerica will maintain documentation of the purity/cleanliness for these materials. The TestAmerica QA Officers are ultimately responsible for ensuring that these materials are acceptable for the project. The acceptability of these materials for use will be evaluated by reviewing lot analysis certificates (deionized water, chemical preservatives, and containers). Purge-and-trap

grade water will be obtained from TestAmerica's volatile organic analysis laboratory and will meet the acceptability requirements for method blank samples specified in their VOC analysis SOP. Water, preservatives, and containers that do not meet TestAmerica's acceptability requirements will not be shipped to the field.

K.5.2 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

The procedures for sample handling, labeling, shipping, and chain-of-custody documentation are provided in the subsections that follow.

K.5.2.1 SAMPLE HANDLING

The procedures used to collect the samples are provided in Appendix J-J of the FSP. Sample aliquots will be containerized in order of decreasing analyte volatility. Table K.5.2 identifies the requirements for the number of containers, container volume, container type (material of construction), preservation, holding time periods, packaging, and shipping for the analyses associated with each sampling program.

The sample numbering system for the project has been designed to uniquely identify each sample from each sampling program and event. This numbering system consists of the sample matrix code, project reference number, sample collection date, sampler's initials, and sequential number beginning with 001 continuing throughout the sampling program and event.

An example of the sample numbering system follows:

MC-38443-mmddyy-XX-001

where:

MC (Matrix Code)	=	GW - groundwater, SW - surface water, S – soil, SG - soil gas, SE-sediment, W-field blank samples
38443	=	Project reference number
mmddyy	=	Date in month/day/year
XX	=	Sampler's first and last initials
001	=	Sequential number for event

Field duplicate samples will be numbered consistent with this system to avoid laboratory bias of field QC samples. Samples designated for MS/MSD analysis will be identified as such in the remarks column of the chain-of-custody form. Trip blank samples are provided by the laboratory and labeled as such. Trip blank samples will be identified on the chain-of-custody form with the date of collection (Trip Blank-mm/dd/yy) to ensure that the trip blank sample data are uniquely identified.

Samples collected for off-Site analysis will be placed in shipping coolers containing bagged, cubed ice immediately following collection. The samples will be grouped in the shipping cooler by the order in which the samples are collected, and shipped to the laboratory via an overnight courier service, generally on the day they are collected. The only exceptions to this procedure will be for samples collected after the courier service has picked up the shipment for the day (generally only at remote sites) and samples collected on a Sunday or holiday. In these instances, the samples will be shipped on the next business day. An example shipping form is provided in Appendix K-H.

The laboratory will group the samples in sample delivery groups (SDGs) by sampling program. An SDG is a group of field samples (including field QC samples) received by the laboratory within seven calendar days.

K.5.2.2 SAMPLE CUSTODY

Chain-of-custody is the sequence of possession of an item. An item (such as a sample or final evidence file) is considered to be in custody if the item is in actual possession of a person, the item is in the view of the person after being in his/her actual possession, or the item was in a person's physical possession but was placed in a secure area by that person. Field, laboratory, and final evidence files custody procedures are described in the subsections that follow.

K.5.2.2.1 FIELD CUSTODY PROCEDURES

Logbooks will used to record field data collection activities. Entries into field logbooks will be described in as much detail as possible to ensure that a particular situation could be reconstructed solely from logbook entries. Field logbooks will be bound field survey books or notebooks with consecutively numbered pages. Logbooks will be assigned to field personnel and will be stored at CRA's Detroit, Michigan office when not in use. Each logbook will be identified by the project-specific document number (38443).

The title page of each logbook will contain the following information:

- person to whom the logbook is assigned;
- logbook number;
- project name;
- project start date; and
- end date.

Entries into the logbook will contain a variety of information. At the beginning of each day's logbook entry, the date, start time, weather, names of all sampling team members present, and the signature of the person making the entry will be entered. The names of individuals visiting the site or field sampling team and the purpose of their visit will also be recorded in the field logbook.

All field measurements obtained and samples collected will be recorded. All logbook entries will be made in ink, signed, and dated with no erasures. If an incorrect logbook

entry is made, the incorrect information will be crossed out with a single strike mark, which is initialed and dated by the person making the erroneous entry. The correct information will be entered into the logbook adjacent to the original entry.

Whenever a sample is collected or a measurement is made, a detailed description of the location will be recorded in the logbook. Photographs taken at a location, if any, will also be noted in the logbook. All equipment used to obtain field measurements will be recorded in the field logbook. In addition, the calibration data for all field measurement equipment will be recorded in the field logbook or on standard field forms.

Samples will be collected following the sampling procedures documented in the Appendix J-J of the FSP. The equipment used to collect samples, time of sample collection, sample description, volume and number of containers, preservatives added (if applicable) will be recorded in the field logbook. Each sample will be uniquely identified using the sample numbering system provided in Section K.5.2.1 of this QAPP.

The sample packaging and shipping procedures summarized below will ensure that the samples arrive at the laboratory with the chain-of-custody intact:

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred to another person or the laboratory. As few people as possible will handle the samples.
2. All sample containers will be identified by using sample labels that include the sample identification number, sample type, sampler, date of collection and analyses to be performed. Sample labels will be completed for each sample using waterproof ink. An example of a sample label is provided in Appendix K-H.
3. Samples will be accompanied by a properly completed chain-of-custody form. The sample identification numbers and required analyses will be listed on the chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving the samples will sign and record the date and time on the form. The chain-of-custody form documents sample custody transfers from the sampler to another person, to the laboratory, or to/from a secure storage area.
4. Samples will be properly packaged for shipment (see Table K.5.2) and dispatched to the laboratory for analysis with a separate signed chain-of-custody

form enclosed in and secured to the inside top of each shipping cooler. Shipping coolers will be secured with custody tape for shipment to the laboratory. The custody tape is then covered with clear plastic tape to prevent accidental damage to the custody tape. An example of the custody tape to be used for this project is provided in Appendix K-H.

5. If samples are collocated with a government agency or other entity, it is the responsibility of that entity to prepare its own chain-of-custody form for the samples. Information regarding the identity of the entity and the samples that are being collocated will be recorded in the field logbook.
6. All sample shipments will be accompanied by the chain-of-custody form identifying its contents. The chain-of-custody form is a four-part carbonless-copy form. The form is completed by the sampling team, which, after signing and relinquishing custody to the shipper, retains the bottom (goldenrod) copy. The shipper, if different than the sampling team members, retains the pink copy after relinquishing custody to the laboratory. The yellow copy is retained by the laboratory and the fully executed white copy is returned as part of the data deliverables package. An example chain-of-custody form is provided in Appendix K-H.
7. If the samples are sent by common carrier, a bill of lading (e.g., FedEx airbill) will be used and copies will be retained as permanent documentation. Commercial carriers are not required to sign the chain-of-custody form as long as the form is sealed inside the sample cooler and the custody tape remains intact.

K.5.2.2.2 LABORATORY CUSTODY PROCEDURES

Laboratory sample custody begins when the samples are received at the laboratory. TestAmerica's sample receiving group will assign a unique laboratory sample identification number to each incoming sample. The field sample identification numbers, laboratory sample identification numbers, date and time of sample collection, date and time of sample receipt, and requested analyses will be entered into the sample receiving log. TestAmerica's sample log-in, custody, and document control procedures are detailed in the appropriate SOPs in Appendix K-F.

Following log-in, all samples will be stored within an access-controlled location and will be maintained properly preserved (as defined in Table K.5.2) until completion of all

laboratory analyses. Unused sample aliquots and sample extracts/digestates/distillates will be maintained properly preserved for a minimum of 60 days following receipt of the final report by CRA. TestAmerica will be responsible for the disposal of unused sample aliquots, sample containers, and sample extracts/digestates/distillates in accordance with all applicable local, state, and federal regulations. Sample tags will be retained by the TestAmerica until completion of the analysis and shall be returned to CRA with the laboratory final analytical report.

The laboratory will be responsible for maintaining analytical logbooks and laboratory data. Raw laboratory data files will be inventoried and maintained by the laboratory for a period of five years, at which time CRA will advise the laboratory regarding the need for additional storage.

K.5.2.2.3 FINAL EVIDENCE FILES CUSTODY PROCEDURES

The final evidence file for the project will be maintained by CRA and will consist of the following:

1. project plan;
2. project logbooks;
3. field data records;
4. sample identification documents;
5. chain-of-custody records;
6. correspondence;
7. references, literature;
8. final data packages;
9. miscellaneous - photos, maps, drawings, etc.; and
10. final report.

The final evidence file materials will be the responsibility of the evidentiary file custodian (CRA's Project Manager) with respect to maintenance and document removal. All records will be maintained for a period of six (6) years following completion of the 10-year maintenance period as noted in Section K.4.4.3. USEPA is to be notified 90 days

prior to disposal or destruction of records after the six-year maintenance period following completion of the remedial action has expired.

K.5.3 ANALYTICAL METHOD REQUIREMENTS

The field and laboratory analytical methods that will be used during the investigative activities are detailed in the following subsections.

K.5.3.1 FIELD ANALYTICAL METHODS

Field analytical procedures include the measurement of pH/temperature, specific conductivity, turbidity, dissolved oxygen, and oxidation/reduction potential (ORP) during sampling of groundwater at the Site. Specific guidance in the measurement of these parameters is presented in the SOPs provided in Appendix K-F.

K.5.3.2 LABORATORY ANALYTICAL METHODS

All samples will be analyzed by TestAmerica-NC with the exception of the soil gas samples which will be analyzed by TestAmerica-LA, and the asbestos samples which will be analyzed by subcontracted EML San Bruno laboratory. In general, water and soil samples will be acid digested and the digestates analyzed for metals by several instrumental methods including inductively coupled plasma (ICP) emission spectroscopy, ICP-Mass Spectroscopy (MS) and cold vapor atomic absorption (CVAA) spectroscopy. Water and soil samples are analyzed for TCL VOC by purge and trap GC/MS. Semi-volatile organics (SVOC, PCBs, Pesticides and Herbicides) are solvent extracted and the extracts are analyzed by GC with electron capture detection (ECD) for PCBs and pesticides and MS for the SVOC. Dioxin and furans are spiked with isotopically labeled dioxin and furans and then solvent extracted and the extracts are analyzed by high resolution (HR) GC/HRMS. Methane, ethane, and ethene are analyzed as dissolved gases by headspace gas chromatography (GC). The remaining inorganic parameters are analyzed by various gravimetric, colorimetric, microscopic, and spectrophotometric procedures. The concentration of asbestos in wipe samples will be visually estimated using EPA Method 600/R-93/116. In general, soil samples will be

crushed and analyzed for asbestos by performing a 400-point count technique which has a detection limit of 0.25%, under California Air Resource Board 435 method.

The analytical methods that will be used by TestAmerica for analyzing the project samples are presented in Table K.5.3. TestAmerica's SOPs for the analytical methods are presented in Appendix K-F. Method validation and detection limit study information for the analyses are included in TestAmerica's SOPs.

The quantities and types of QC samples for the investigation program are included in Table K.3.1.

K.5.4 QUALITY CONTROL REQUIREMENTS

The field and laboratory QC requirements for the investigative activities are discussed in the following subsections. Specific QC checks employed and frequency of analyses are provided in the field and laboratory SOPs in Appendix K-F.

K.5.4.1 FIELD SAMPLING QUALITY CONTROL

Field QC requirements include analyzing reference standards for instrument calibration and for routine calibration checks. The acceptance criteria are provided in the SOP in Appendix K-F. Field QC samples for this project include field duplicate samples to assess the overall precision of the sampling and analysis event and trip blank samples to monitor cross-contamination of samples by VOCs. The frequency of collection of these field QC samples were provided in Section K.4.2 and Table K.3.1 of this QAPP. The evaluation of field QC data is provided in Section K.5.9.2 of this QAPP.

K.5.4.2 ANALYTICAL QUALITY CONTROL

The laboratory QC requirements for TCL VOC analyses to be performed on Site samples include analyzing mass tuning standards, method blanks, instrument blanks, initial calibration standards, continuing calibration verification standards, surrogate standards, MS/MSDs, and LCSs. The acceptance criteria for all these QC checks except MS/MSD samples, surrogates, and LCSs are in TestAmerica's SOPs.

The laboratory QC requirements for the methane analyses to be performed on Site samples include analyzing method blanks, initial calibration verification standards, continuing calibration verification standards, surrogate standards, MS/MSD samples, and LCSs. The analysis frequency for these QC samples are included in the applicable TestAmerica SOP in Appendix K-F. The acceptance criteria for all these QC checks except MS/MSD samples, surrogates, and LCSs are in TestAmerica's SOPs.

The laboratory QC requirements for metals analyses to be performed on Site samples include analyzing preparation blanks, initial calibration blanks, continuing calibration blanks, initial calibration verification standards, continuing calibration verification standards, interference check standards, internal standards, serial dilution samples, MS/MSD samples, and LCSs. The analysis frequency for these QC samples are included in the applicable TestAmerica SOPs in Appendix K-F. The acceptance criteria for all these QC checks except MS/MSD samples and LCSs are in TestAmerica's SOPs.

The laboratory QC requirements for inorganic analyses to be performed on Site samples include analyzing method blanks, initial calibration standards, calibration check standards, MS/MSDs (if applicable), and LCSs. The acceptance criteria for all these QC checks except MS/MSD samples, surrogates, and LCSs are in TestAmerica's SOPs.

Laboratory QC batch control analyte MS/MSD and LCS acceptance criteria are provided in Table K.4.3 of this QAPP. The acceptance criteria for surrogates are provided in Table K.4.4. These acceptance criteria and the acceptance criteria for "all analyte" QC checks are included in the TestAmerica Reference Data Summary provided in Appendix K-G. The QC acceptance criteria and the MDLs included in this QAPP are updated by the laboratory on a periodic basis. The acceptance criteria in effect when the samples are analyzed will be identified in the laboratory final analytical reports, which may be different than those identified in the QAPP.

K.5.5 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

The procedures used to verify that instruments and equipment are functional and properly maintained are described in the following subsections.

K.5.5.1 FIELD INSTRUMENT MAINTENANCE

The field equipment for this project includes flow-through cell type water quality meters and PIDs. Specific preventive maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. The maintenance schedule and trouble-shooting procedures for field instruments are presented in Table K.5.1.

K.5.5.2 LABORATORY INSTRUMENT MAINTENANCE

As part of their QA/QC program, the laboratories conduct routine preventive maintenance program to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees will regularly perform routine scheduled maintenance and repair of (or coordinate with the instrument manufacturer for the repair of) all instruments. All maintenance that is performed will be documented in the laboratory's maintenance logbooks. All laboratory instruments are maintained in accordance with manufacturer's specifications.

Table K.5.1 provides examples of the frequency at which components of key analytical instruments or equipment will be serviced. The SOPs in Appendix K-F provide complete details for instrument preventive maintenance.

K.5.6 CALIBRATION PROCEDURES AND FREQUENCY

The procedures for maintaining the accuracy for all the instruments and measuring equipment which are used for conducting field tests and laboratory analyses are described in the following subsections. These instruments and equipment will be calibrated prior to each use or according to a periodic schedule.

K.5.6.1 FIELD INSTRUMENTS/EQUIPMENT

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and

reproducibility of results are consistent with the manufacturer's specification and requirements presented in the SOPs in Appendix K-F.

Equipment to be used during field sampling will be examined to confirm that it is in operating condition. This includes checking the manufacturer's operating manual for each instrument to ensure that all maintenance requirements are being observed. Individual calibration records for each field instrument that will be used for the project will be reviewed to ensure that any prior equipment problems have not been overlooked and all necessary repairs to equipment have been completed.

K.5.6.2 LABORATORY INSTRUMENTS

Calibration of laboratory equipment will be based on approved written procedures. Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing quality control activities. These records will be filed at the location where the work is performed and will be subject to QA audit. For all instruments, the laboratory will maintain a properly trained repair staff with in-house spare parts or will maintain service contracts with vendors.

The records of calibration will be kept as follows:

1. If possible, each instrument will have record of calibration permanently affixed with an assigned record number.
2. A logbook will be assigned to each instrument showing description, manufacturer, model numbers, date of last calibration and the signature of the person who calibrated the instrument, due date of next calibration and compensation or correction figures, as appropriate.
3. A written stepwise calibration procedure will be available for each piece of test and measurement equipment.
4. Any instrument that is not calibrated to the manufacturer's original specification will display a warning tag or will otherwise be removed from service, as appropriate.

Specific calibration procedures and frequencies are detailed in the laboratory SOPs in Appendix K-F.

K.5.7 INSPECTION/ACCEPTANCE CRITERIA FOR SUPPLIES AND CONSUMABLES

The procedures that will be used to ensure that supplies and consumables used in the field and laboratory will be available as needed and free of contaminants are detailed in the following subsections.

K.5.7.1 FIELD SUPPLIES AND CONSUMABLES

Supplies and consumables for field measurements and sampling will be obtained from various vendors and include reference standards and solutions for pH, specific conductance, turbidity, dissolved oxygen and ORP, sample containers, preservatives, and detergent and water for equipment decontamination. The vendors and inspection and acceptance criteria for these field supplies were presented in Section K.5.1.4 of this QAPP. Additional field supplies and consumables include pump tubing, personnel protective equipment (PPE). Pump tubing will be constructed of pre-cleaned high-density polyethylene. These materials will not introduce contaminants into the samples or interfere with the analyses. All field supplies will be consumed or replaced with sufficient frequency to prevent deterioration or degradation that may interfere with the analyses.

K.5.7.2 LABORATORY SUPPLIES AND CONSUMABLES

TestAmerica's vendor for general labware and reagents is Fisher Scientific. Vendors for chromatography supplies and organic standards include Ultra Scientific, Supelco, Accustandard, Restek, ChemService, and Aldrich Chemical. Vendors for metals and general chemistry parameters supplies and standards include Ultra Scientific, High Purity Standards, and Inorganic Ventures. The lot numbers of reagents and standards are recorded and dates of receipt, first use, and expiration are documented. Certificates of analysis are maintained on file to document reagent/standard purity.

The SOPs in Appendix K-F provide details on identifying contaminants in reagents and standards, determining deterioration of reagents and standards, and the corrective

actions required if contaminants or deterioration are identified. The laboratory QA Officer is ultimately responsible for the ensuring the acceptability of supplies and consumables.

K.5.8 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

Historical data for the Site were generated during the various studies and monitoring events identified in Appendix K-A.

K.5.9 DATA MANAGEMENT

The procedures for managing data from generation to final use and storage are detailed in subsections that follow.

K.5.9.1 DATA RECORDING

Field data will be recorded in field logbooks and consist of measurements from direct reading instruments or direct measurements. Field staff are responsible for recording field data and the Field QA Officer is responsible for identifying and correcting recording errors.

Laboratory data are recorded in a variety of formats. Data from instruments are recorded on magnetic media, strip charts, or bench sheets. The laboratory SOPs in Appendix K-F provide the data-recording requirement for each preparation and analysis method.

K.5.9.2 DATA VALIDATION

Validation of field data for this project will primarily consist of checking for transcription errors and review of data recorded in field logbooks. Data transcribed from the field logbook into summary tables for reporting purposes will be verified for

correctness by the Field QA Officer or his designee. Any limitations on the use of field data will be included in the investigative activity reports.

Validation of the analytical data will be performed by CRA chemistry staff under the direction of CRA's QA Officer. Data evaluation SOPs will be based on the following:

- QAPP requirements;
- Laboratory SOPs;
- the relevant and applicable evaluation criteria outlined in "USEPA CLP NFG for Superfund Organic Methods Data Review" (July 2007);
- the relevant and applicable evaluation criteria outlined in "USEPA CLP NFG for Inorganic Data Review" (October 2004) (National Functional Guidelines); and
- CRA's "Analytical Data Quality Assessment and Validation SOP" (April 2008), provided in Attachment K-J.

The evaluation and action criteria specified in these documents will be used for validating the data. However, the acceptance limits for QC data will be the control limits determined statistically by the laboratory, not the control limits specified in the National Functional Guidelines. Qualifiers assigned to the data will be consistent with the data qualifiers specified in the National Functional Guidelines.

Analytical data will be validated at one of two levels depending on the sampling event and data quality objectives. The elements reviewed under these two data validation levels are described in the following sections and in Table K.5.4. All samples evaluated for the Human Health Risk Assessment and the Ecological Risk Assessment will undergo a full data validation with the exception of samples collected for waste characterization, soil gas analysis, and vertical aquifer sampling which will undergo a reduced data validation.

The following deliverables will be evaluated for all samples (reduced data validation):

i) technical holding times;

ii) blanks;

iii) system monitoring compounds (surrogate spikes);

iv) MS/MSD results;

v) laboratory control samples; and

vi) field duplicates.

A minimum of ten percent of all data will undergo a raw data review including chromatography and mass spectral data review, calculation checks from sample preparation though to final data, and a review for transcription errors. The following deliverables will be evaluated during full validation:

Organic Analyses:

i) technical holding times;

ii) GC/MS instrument performance check;

iii) initial and continuing calibration;

iv) blanks;

v) system monitoring compounds (surrogate spikes);

vi) internal standard performance;

vii) MS/MSD results;

viii) laboratory control samples;

ix) field duplicates

x) target compound identification and quantitation; and

xi) system performance

Inorganic Analyses:

i) technical holding times;

ii) initial and continuing calibration standards and blanks;

iii) ICP/MS internal standard performance;

iv) Blanks

v) interference check samples;

vi) laboratory control samples;

vii) MS/MSD results;

viii) Post digestion spikes;

ix) ICP serial dilution;

x) Analyte identification and quantitation; and

xi) Field duplicates.

The results of the data validation process will be documented in a memorandum that specifies all limitations on the usability of the analytical data.

K.5.9.3 DATA TRANSFORMATION/DATA REDUCTION

Field data reduction procedures will be minimal in scope compared to those implemented for laboratory data. Only direct reading instrumentation will be employed in the field. The use of field instrument meters will generate data read directly from the meters following calibration as outlined in the SOPs in Appendix K-F. These data will be recorded into field logbooks immediately after the measurements are taken.

Laboratory data reduction procedures will be followed according to the following protocol:

1. Raw data produced and checked by the responsible analyst is turned over for independent review by another analyst.
2. The area supervisor or senior chemist reviews the data for attainment of quality control criteria established by the QAPP.
3. The area supervisor will decide whether any sample re-analysis is required.
4. Upon completion of all reviews and acceptance of the raw data by the area supervisor, a report will be generated and sent to the laboratory Project Manager.
5. The laboratory Project Manager will complete a thorough inspection of all reports.

6. Following review and approval of the preliminary report by the laboratory Project Manager, final reports will be generated and signed by the laboratory Project Manager.

Specific equations used for data reduction are contained in the SOPs in Appendix K-F.

K.5.9.4 DATA TRANSMITTAL/TRANSFER

Field data from surveying and water level measurements will be entered into a standard Microsoft Excel spreadsheet format. CRA's Field QA Officer is responsible for verifying the correctness of the field data after the data are transferred to a spreadsheet format. The geographical data are maintained in a database that is described below.

TestAmerica will provide electronic data deliverables (EDDs) in the EQuIS 4-file format. EQuIS is a database product from EarthSoft that uses Microsoft Access as the database engine. The laboratory data are downloaded into the EDDs directly from the laboratory information management system (LIMS), thus eliminating the possibility of manual transcription errors. The EDDs are imported with EQuIS and the data are maintained in the database for manipulation and presentation.

CRA's QA Officer is responsible for verifying the correctness of the analytical database after the laboratory data for each event have been imported. This is accomplished by comparing the data from the database to the hardcopy analytical reports for a minimum of 10 percent of the sample results. If discrepancies between the database and hardcopy analytical reports are detected, a complete verification of the database will be performed or a new EDD will be submitted, imported, and verified as described previously.

K.5.9.5 DATA ANALYSIS

The data from the O&M groundwater monitoring will be compared to the State generic clean-up criteria to evaluate the progression of MNA at the Site.

K.5.9.6 DATA ASSESSMENT

Assessment of laboratory data by TestAmerica will be performed using the procedures detailed in the SOP entitled "Statistical Evaluation of Data and Control Charts", which is provided in Appendix K-F. Specific data assessment for each analytical method is provided in TestAmerica's SOPs in Appendix K-F. These assessments included determining the mean, standard deviation, relative standard deviation, percent difference, RPD, and percent recovery for certain QC elements.

Assessment of QC data for data validation purposes will include determining the percent recovery, RPD, and percent completeness. The statistical equations to determine these parameters are provided in Section K.7.2 of this QAPP.

K.5.9.7 DATA TRACKING

Data generated in the field, such as water level measurements, will be recorded in field logbooks. Survey data will be generated by the surveying subcontractor and provided to CRA. There are no unique or special tracking requirements for these data. The data will be transcribed for analysis and reporting as discussed in Section K.5.9.4, and the original survey data and field logbooks will be maintained in the final evidence file.

Laboratory data tracking procedures are provided in the SOPs in Appendix K-F. These SOPs provide the procedures for tracking data from generation to reporting. TestAmerica's LIMS also provides a means for tracking data in the laboratory. The laboratory Operations Manager is ultimately responsible for data tracking in the laboratory.

Tracking of analytical data in the EQuIS database includes recording the laboratory generating the data, the date when EDD was received and imported, the date when qualifiers were applied to the results, and the level of data validation performed. CRA's Project Manager is ultimately responsible for tracking data from entry into the database to reporting.

K.5.9.8 DATA STORAGE AND RETRIEVAL

Laboratory data will be stored by TestAmerica in hardcopy format at their North Canton, Ohio facility. Data are archived on site for a period of 5 years, after which time the data are warehoused off site. Electronic instrument data are maintained on magnetic media (i.e., magnetic tape, compact disc, etc.) for this same time-period. TestAmerica's records manager is Lance Hershman, who is responsible for data archiving and retrieval at the North Canton, Ohio facility.

CRA's Project Manager is responsible for project data storage and retrieval. Field logbooks will be maintained in CRA's Detroit, Michigan office. At the conclusion of the soil investigation, field logbooks associated with this task will be archived at CRA's Waterloo, Ontario, Canada headquarters. Upon completion of the remedial action, the final evidence file will be archived at CRA's Waterloo, Ontario, Canada headquarters.

K.5.9.9 DATA SECURITY

Laboratory data security is the responsibility of TestAmerica's records manager. Archived data cannot be accessed without authorization and the name and purpose of personnel accessing archived data are recorded. TestAmerica's LIMS is password protected and access rights are restricted by job function.

CRA's data security procedures include limiting project database access to database analysts and general building security procedures.

K.6.0 ASSESSMENT/OVERSIGHT

The following subsections describe the procedures used to ensure proper implementation of this QAPP and the activities for assessing the effectiveness of the implementation of the project and associated QA/QC activities.

K.6.1 ASSESSMENTS AND RESPONSE ACTIONS

Assessments consisting of internal and external audits may be performed during the project. Internal technical system audits of both field and laboratory procedures will be conducted to verify that sampling and analysis are being performed in accordance with the procedures established in the Appendix J-J of the FSP and Appendix K-F of the QAPP. External field and laboratory audits may be conducted by USEPA and the OEPA.

An internal field technical system audit of field activities, including sampling and field measurements, will be conducted by the Field QA Officer or his designee at the beginning of the field sampling activities to identify deficiencies in the field sampling and documentation procedures. The field technical system audit will include examining field-sampling records, field instrument operating records, field instrument calibration records, and chain-of-custody documentation. In addition, sample collection, handling, and packaging in compliance with the established procedures will be reviewed during the field audit. Any deficiencies identified will be documented and corrective actions will be taken to rectify the deficiencies.

Corrective action resulting from internal field technical system audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The Field QA Officer will identify deficiencies and recommended corrective action to the Project Manager. Implementation of corrective actions will be performed by the Field QA Officer and field team. Corrective action will be documented in the field logbook and/or the project file. Follow-up audits will be performed as necessary to verify that deficiencies have been corrected, and that the QA/QC procedures described in this QAPP and the Appendix J-J of the FSP are maintained throughout the project.

An external field technical system audit may be conducted by USEPA Region 5 FSS any time during the field operations. These audits may or may not be announced and are conducted at the discretion of USEPA Region 5.

An internal laboratory technical system audit will be conducted by the TestAmerica QA Officer or designee. The laboratory technical system audit is conducted on an annual basis and includes examining laboratory documentation regarding sample receiving, sample log-in, storage and tracking, chain-of-custody procedures, sample preparation and analysis, instrument operating records, data handling and management, data tracking and control, and data reduction and verification. The laboratory QA Officer will evaluate the results of the audit and provide a final report to section managers and the Laboratory Operations Manager that includes any deficiencies and/or noteworthy observations.

Corrective action resulting from deficiencies identified during the internal laboratory technical system audit will be implemented immediately. The Operations Manager or section leaders, in consultation with the laboratory supervisor and staff, will approve the required corrective action to be implemented by the laboratory staff. The laboratory QA/QC Officer will ensure implementation and documentation of the corrective action. All problems requiring corrective action and the corrective action taken will be reported to the laboratory Project Manager. Follow-up audits will be performed as necessary to verify that deficiencies have been corrected, and that the QA/QC procedures described in the QAPP are maintained throughout the project.

An external laboratory audit may be conducted by USEPA Region 5 FSS or OEPA personnel. These audits may or may not be announced and are at the discretion of USEPA Region 5. The external laboratory audits will include, but not be limited to, reviewing laboratory analytical procedures, laboratory on-site audits, and/or submitting performance evaluation samples to the laboratory for analysis.

An external laboratory audit may be conducted at least once prior to the initiation of the sampling and analysis activities.

K.6.2 REPORTS TO MANAGEMENT

Quality Assurance Management Reports will be prepared during the investigative activities. These QA Management Reports will be included with the investigative activity progress reports that are submitted to USEPA and OEPA when data gathering or assessment activities are being conducted. Minimally, these reports will include project status, results of performance evaluations and system audits, results of periodic data quality validation and assessment and data use limitations, and any significant QA problems identified and corrective actions taken.

CRA's QA Officer will be responsible within the organizational structure for preparing these reports. CRA's Project Manager will be provided with these reports for distribution with monthly status reports. The investigative activity reports and technical memoranda will also include a separate QA/QC section that will summarize data quality information contained in the periodic QA Management Reports and provides an overall data quality assessment compared to the data quality objectives outlined in this QAPP.

K.7.0 DATA VERIFICATION/VALIDATION AND USABILITY

The QA activities that will be performed to ensure that the investigative activity data are scientifically defensible, properly documented, of known quality, and meet the project objectives are described in the following sections.

K.7.1 DATA REVIEW, VERIFICATION, AND VALIDATION REQUIREMENTS

All field and laboratory data will be reviewed and verified/validated. The procedures and criteria used to verify and validate field and laboratory data will consist of evaluating the data to the measurement performance criteria in Section K.4.2 of this QAPP. Field data and logbooks will be reviewed to ensure that the requirements of the sampling program, including the number of samples and locations, sampling procedures, and sample handling, were fulfilled. Acceptable departures from the planned sampling program, such as collecting a sample from an adjacent location because of a subsurface obstruction, will not impact the data usability.

Sample collection procedures will be reviewed for compliance with the requirements of the Appendix J-J of the FSP and QAPP. If alternate sampling procedures were used, the acceptability of the procedure will be evaluated to determine the affect on the usability of the data. Data usability will not be affected if the procedure used is determined to be an acceptable alternative that fulfills the measurement performance criteria in Section 4.2 of this QAPP. Acceptable alternate sampling procedures include collecting soil samples with a drill rig instead of a direct-push sampling device and using a submersible pump instead of a bladder pump to collect groundwater samples. However, data generated from sampling procedures that do not provide representative samples will be rejected. An example would be a groundwater sample collected from a monitoring well that was not properly purged prior to sampling.

Sample handling records will be reviewed to ensure that sample integrity remained intact from collection to laboratory receipt and that samples were properly preserved. Chain-of-custody documentation and sample condition upon laboratory receipt will be reviewed. The data from samples for which the chain-of-custody or sample identification cannot be verified will be rejected. The data for samples that were not properly preserved will be qualified or rejected depending on the severity of the

deviation from the requirements of the Appendix J-J of the FSP and Appendix K-F of the QAPP. The criteria for rejecting improperly preserved samples will be that the sample has been rendered unsuitable for analysis. An example of this situation is preserving a water sample designated for cyanide analysis with acid. If minor pH adjustments are required at the laboratory to account for sample buffering affects, data qualification may be required. The criteria for qualifying or rejecting data for samples that are received at the laboratory without being properly preserved, but not rendered unsuitable for analysis, will be based on the sample holding time period evaluation criteria for unpreserved samples specified in the National Functional Guidelines. Data qualification will be consistent with the action specified in the National Functional Guidelines.

Field and laboratory data will be verified to ensure that the methods used to analyze the samples were consistent with the requirements of this QAPP. Data generated from the use of unapproved methods will be rejected. Acceptable departures from the methods and SOPs specified in this QAPP include using an alternate field meter of comparable capability if the specified meter becomes inoperable.

QC data will be reviewed to determine compliance with the acceptance criteria in Section K.5.4 of this QAPP. QC data that do not meet the acceptance criteria will result in sample data qualification. Significant departures from the QC acceptance criteria may result in rejected data. Situations that result in data rejection include samples analyzed beyond twice the technical holding time period, internal standard recoveries less than 10 percent for non-detected analytes quantitated with that internal standard, surrogate standard recoveries less than 10 percent for non-detected analytes in that sample, inorganic LCS analyte recoveries less than 50 percent if the analyte is not detected in the associated samples, inorganic matrix spike analyte recoveries less than 30 percent if the analyte is not detected in the associated samples, organic matrix spike compound recoveries less than 10 percent if the compound is not detected in the MS/MSD sample, and organic LCS compound recoveries less than 10 percent if the compound is not detected in the associated samples.

K.7.2 VERIFICATION AND VALIDATION METHODS

Field data will be verified by reviewing field documentation and chain-of-custody records. Data from direct-reading instruments will be internally verified by reviewing calibration and operating records. TestAmerica will internally verify the laboratory data

by reviewing and documenting sample receipt, sample preparation, sample analysis (including internal QC checks), data reduction and reporting. Any deviations from the acceptance criteria, corrective actions taken, and data determined to be of limited usability (i.e., laboratory-qualified data) will be noted in the case narrative of the laboratory report.

Data validation will be conducted by CRA's QA personnel consistent with the procedure identified in Section K.5.9.2 of this QAPP. The data verification/validation procedure will identify data as being acceptable, of limited usability (qualified as estimated), or rejected. The conditions that result in data being qualified as estimated or rejected are identified in Section K.7.1 of this QAPP. The results of the data verification/validation will be provided in data validation memoranda that are prepared by CRA's QA Officer.

Data determined to be unusable may require that corrective action to be taken. Potential types of corrective action may include resampling by the field team or reanalysis of samples by the laboratory. The corrective actions taken are dependent upon the ability to mobilize the field team and whether the data are critical for project DQOs to be achieved. Should the CRA QA Officer identify a situation requiring corrective action during data verification/validation, CRA's Project Manager will be responsible for approving the implementation of the corrective action.

K.7.2.1 USABILITY/RECONCILIATION WITH DATA QUALITY OBJECTIVES

The overall usability of the data for the investigative activities will be assessed by evaluating the PARCCS of the data set to the measurement performance criteria in Section K.4.2 of this QAPP using basic statistical quantities as applicable. The procedures and statistical formulas to be used for these evaluations are presented in the following subsections.

K.7.2.2 PRECISION

Project precision will be evaluated by assessing the RPD data from field duplicate samples. Analytical precision will be evaluated by assessing the RPD data from either

duplicate spiked sample analyses or duplicate sample analyses. The RPD between two measurements is calculated using the following simplified formula:

$$RPD = \frac{|R_1 - R_2|}{\left(\frac{R_1 + R_2}{2}\right)} \times 100$$

where:

R_1 = value of first result
R_2 = value of second result

Overall precision for the sampling programs will be determined by calculating the mean RPD for all field duplicates in a given sampling program. This will provide an evaluation of the overall variability attributable to the sampling procedure, sample matrix, and laboratory procedures in each sampling program.

The overall precision requirement will be the same as the project precision. It should be noted that the RPD of two measurements can be very high when the data approach the quantitation limit of an analysis. The calculation of the mean RPD will only include the RPD values for field duplicate sample analyte data that are greater than or equal to 5 times the quantitation limit for an analysis.

K.7.2.3 ACCURACY/BIAS

The data from method/preparation blank samples, trip blank samples, surrogate spikes, MS/MSD samples, and LCSs will be used to determine accuracy and potential bias of the sample data.

The data from method/preparation blank samples provide an indication of laboratory contamination that may result in bias of sample data. Sample data associated with method/preparation blank contamination will have been identified during the data verification/validation process. Sample data associated with method/preparation blank contamination are evaluated during data validation procedure to determine if analytes detected in the samples and the associated method/preparation blanks are "real" or are the result of laboratory contamination. The procedure for this evaluation involves comparing the concentration of the analyte in the sample to the concentration in the

method/preparation blank taking into account adjustments for sample dilutions and dry-weight reporting. In general, the sample data are qualified as not detected if the sample concentration is less than 5 times (10 times for common laboratory contaminants) the method/preparation blank concentration. Typically, the quantitation limit for the affected analyte is elevated to the concentration detected in the sample.

The data from trip blanks provide an indication of field and transportation conditions that may result in bias of sample data. Sample data associated with contaminated trip blank samples will have been identified during the data verification/validation process. The evaluation procedure and qualification of sample data associated with trip blank contamination is performed in the same manner as the evaluation procedure for method blank sample contamination.

Surrogate spike recoveries provide information regarding the accuracy/bias of the organic analyses on an individual sample basis. Surrogate compounds are not expected to be found in the samples and are added to every sample prior to sample preparation/purging. The percent recovery data provide an indication of the effect that the sample matrix may have on the preparation and analysis procedure. Sample data exhibiting matrix effects will have been identified during the data verification/validation process.

Matrix spike sample data provide information regarding the accuracy/bias of the analytical methods relative to the sample matrix. Matrix spike samples are field samples that have been fortified with target analytes prior to sample preparation and analysis. The percent recovery data provide an indication of the effect that the sample matrix may have on the preparation and analysis procedure. Sample data exhibiting matrix effects will have been identified during the data verification/validation process.

Analytical accuracy/bias will be determined by evaluating the percent recovery data of LCSs. LCSs are artificial samples prepared in the laboratory using a blank matrix that is fortified with analytes from a standard reference material that is independent of the calibration standards. LCSs are prepared and analyzed in the same manner as the field samples. The data from LCS analyses will provide an indication of the accuracy and bias of the analytical method for each target analyte.

Percent recovery is calculated using the following formula:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Spiked Sample Result
SR = Sample Result or Background
SA = Spike Added

The percent recovery of LCSs are determined by dividing the measured value by the true value and multiplying by 100.

Overall accuracy/bias for the sampling events will be determined by calculating the percent of accuracy measurements that meet the measurement performance criteria specified in Section K.4.2 of this QAPP. Overall accuracy will be considered acceptable if the surrogate percent recoveries are met for at least 75 percent of the samples and the LCS percent recoveries are met for all the samples and the MS/MSD percent recoveries are met for at least 75 percent of the samples.

K.7.2.4 SAMPLE REPRESENTATIVENESS

Representativeness of the samples will be assessed by reviewing the results of field audits and the data from field duplicate samples. Overall sample representativeness will be determined by calculating the percent of field duplicate sample data that achieved the RPD criteria specified in Section K.4.2 of this QAPP. Overall sample representativeness will be considered acceptable if the results of field audits indicate that the approve sampling methods or alternate acceptable sampling methods were used to collect the samples and the field duplicate RPD data are acceptable for at least 75 percent of the samples.

K.7.2.5 COMPLETENESS

Completeness will be assessed by comparing the number of valid (usable) sample results to the total possible number of results within a specific sample matrix and/or analysis. Percent completeness will be calculated using the following formula:

$$\% \text{ Completeness} = \frac{\text{Number of Valid (usable) measurements}}{\text{Number of Measurements Planned}} \times 100$$

Overall completeness will be assessed by calculating the mean percent completeness for the entire set of data obtained for each sampling program. The overall completeness for the soil investigation will be calculated when all sampling and analysis is concluded. The groundwater and surface water sampling is a long-term program, and the overall completeness will be determined at the conclusion of each monitoring event. Overall completeness will be considered acceptable if at least 90 percent of the data are determined to be valid.

K.7.2.6 COMPARABILITY

The comparability of data sets will be evaluated by reviewing the sampling and analysis methods used to generate the data for each data set. Project comparability will be determined to be acceptable if the sampling and analysis methods specified in this QAPP and any approved QAPP revisions or amendments are used for generating the soil, groundwater, and surface water data.

Overall comparability of data from split samples (samples that are collected at the same time from the same location and split equally between two parties using sample containers from the same source or vendor) will be evaluated by determining the RPD of detected analytes in both samples following data verification/validation. Analytes that are detected in only one of the two samples will be assessed by reviewing the data verification/validation reports for both data sets and determining the cause of the discrepancy. Overall comparability of split sample data will be considered acceptable if the RPD for detected analytes with concentrations greater than or equal to 5 times their respective quantitation limits does not exceed RPD acceptance criteria for field duplicate samples.

K.7.2.7 SENSITIVITY AND QUANTITATION LIMITS

The quantitation limits for the sample data will be reviewed to ensure that the sensitivity of the analyses was sufficient to achieve the generic clean-up criteria for the soil investigation and air monitoring. The method/preparation blank sample data and

LCSs percent recovery data will be reviewed to assess compliance with the measurement performance criteria specified in Section K.4.2 of this QAPP.

Overall sensitivity will be assessed by comparing the sensitivity for each monitoring program (i.e., soil investigation/verification and air monitoring) to the detectability requirements for the analyses. The overall sensitivity for the soil investigation will be assessed when all sampling and analysis is concluded. The groundwater and surface water sampling is a long-term program, and the overall sensitivity will be assessed at the conclusion of each monitoring event. Overall sensitivity will be considered acceptable if quantitation limits for the samples are less than the applicable evaluation criteria.

It should be noted that quantitation limits may be elevated as a result of high concentrations of target compounds, non-target compounds, and matrix interferences (collectively known as sample matrix effects). In these cases, the sensitivity of the analyses will be evaluated on an individual sample basis relative to the applicable evaluation criteria. The need to investigate the use of alternate analytical methods may be required if the sensitivity of the analytical methods identified in this QAPP cannot achieve the evaluation criteria as a result of sample matrix effects.

K.7.2.8 DATA LIMITATIONS AND ACTIONS

Data use limitations will be identified in data quality assessment reports. Data that do not meet the measurement performance criteria specified in this QAPP will be identified and the impact on the project quality objectives will be assessed and discussed in these reports. Specific actions for data that do not meet the measurement performance criteria depends on the use of the data, and may require that additional samples are collected or the use of the data be restricted.

Data quality assessment reports will be prepared at the conclusion of each sampling event. Determination of the overall data quality for a specific sampling program will be conducted at the completion of the program. Data quality assessment reports will be included with the project reports identified in the investigative activities.

K.8.0 REFERENCES

EPA 540/R-93/051. "Specifications and Guidance for Contaminant-Free Sample Containers", EPA 540/R-93/051, 1993.

EPA-540-R-07-003. "USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review", EPA-540-R-07-003, July 2007.

EPA QA/G-5. "EPA Guidance for Quality Assurance Project Plans", EPA QA/G-5, February 1998.

EPA QA/R-5. "EPA Requirements for Quality Assurance Project Plans", EPA QA/R-5, March 2001.

EPA SW-846. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, 3rd Edition with Updates I through III, November 1986.

EPA 540-R-04-004. "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", EPA 540-R-04-004, October 2004.

EPA 2000. "Region 5 Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan, Revision 0", June 2000.

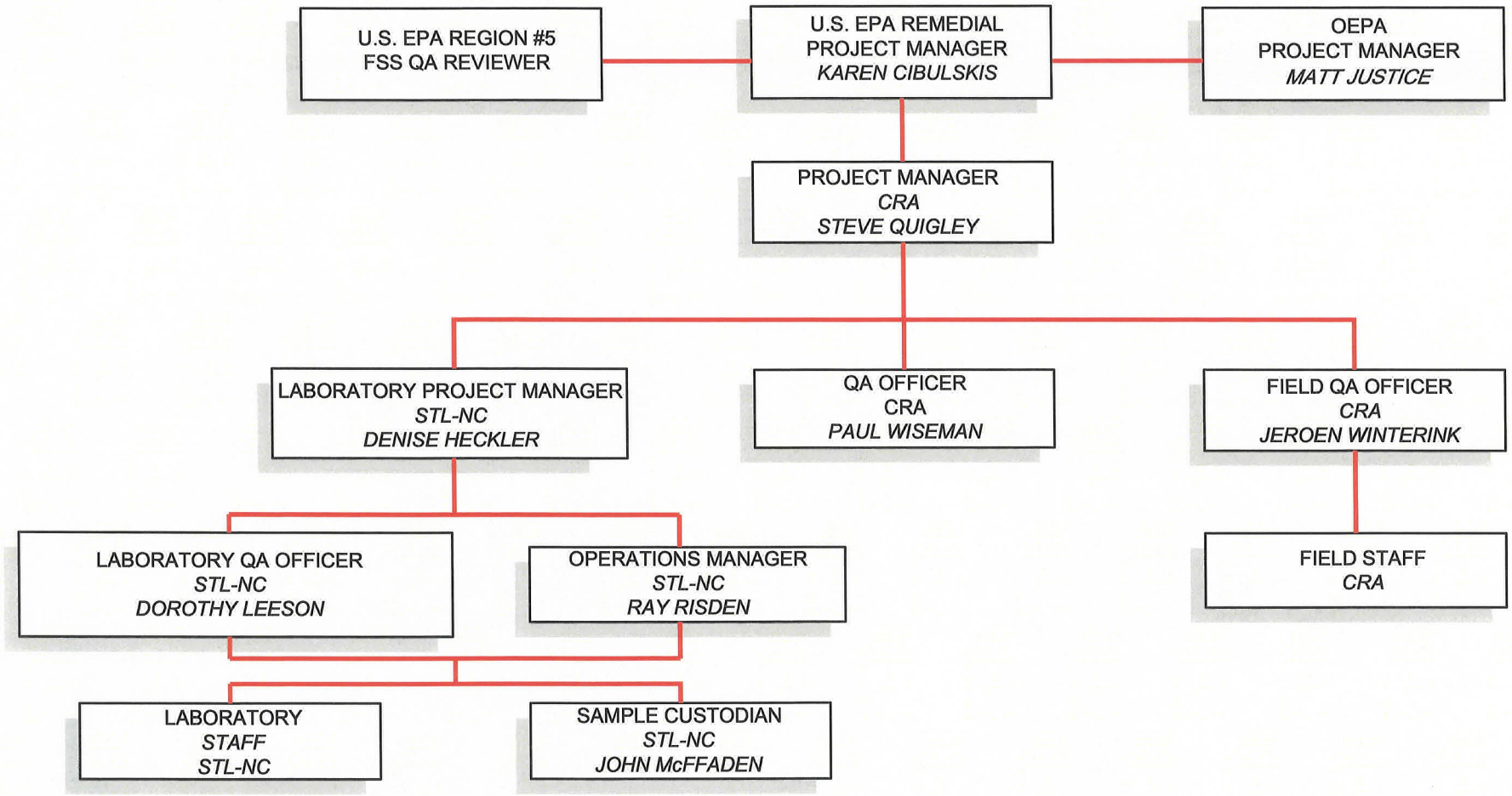
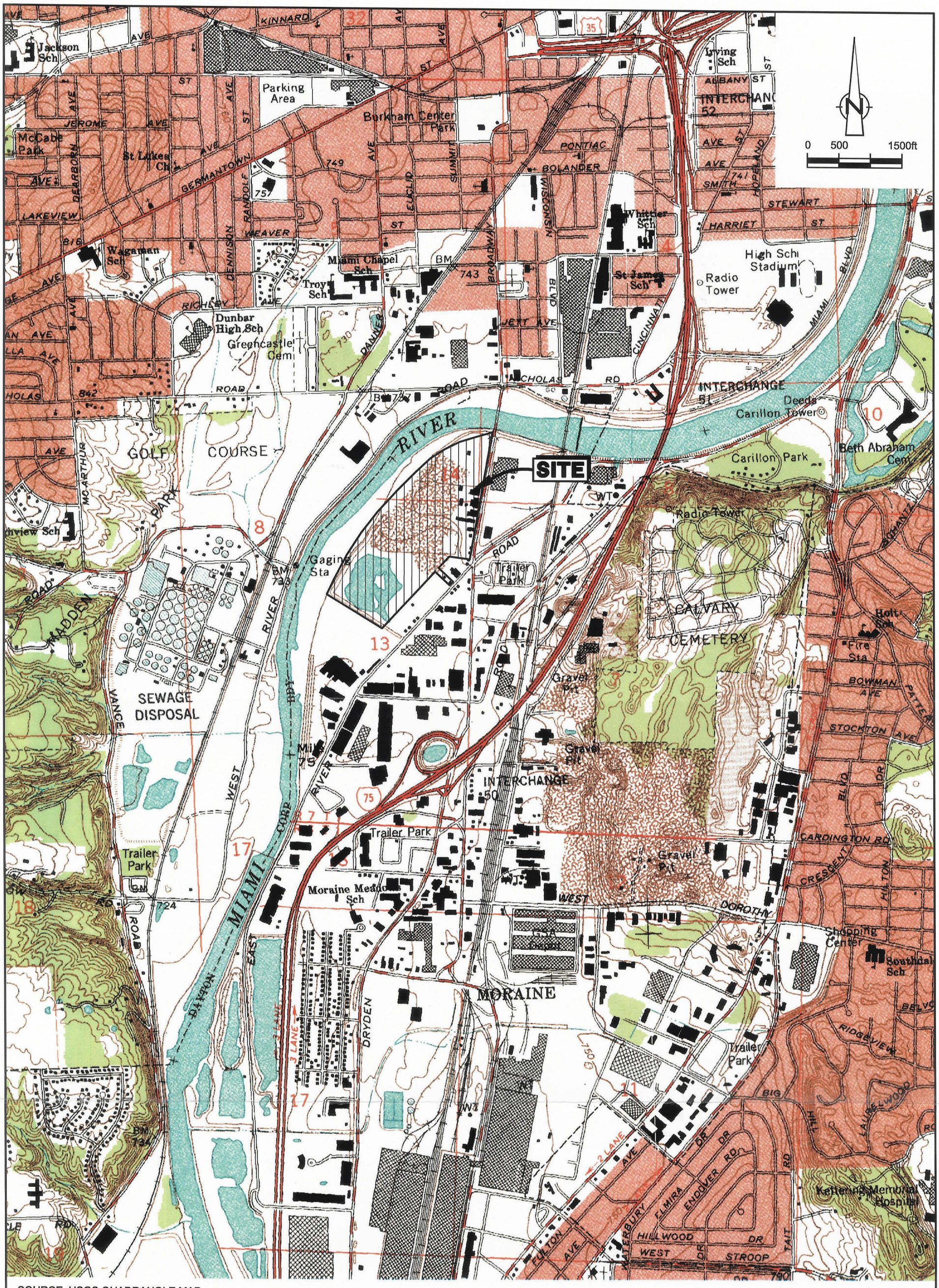


figure K-2.1

PROJECT QA/QC ORGANIZATION
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-07(005)GN-WA005 DEC 19/2006



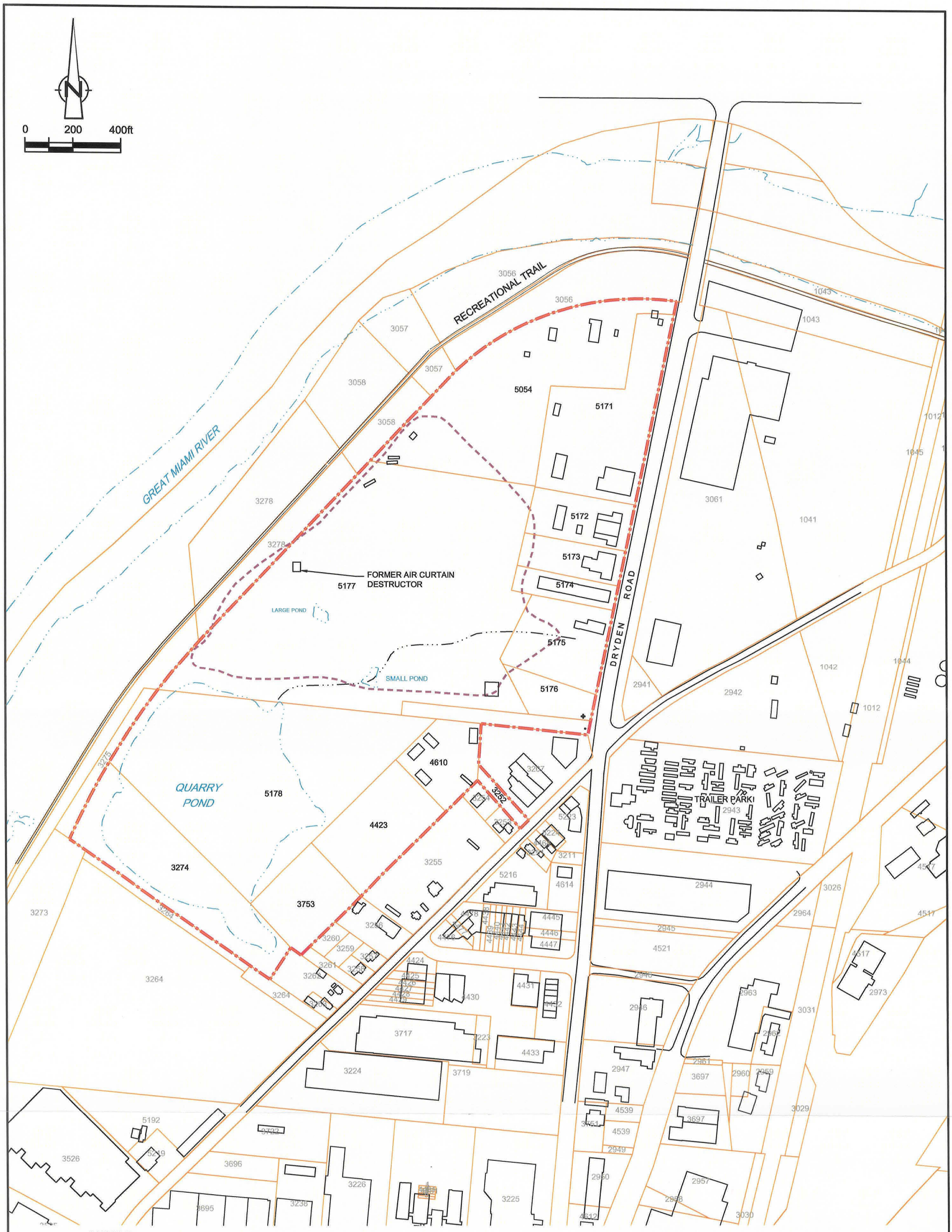
SOURCE: USGS QUADRANGLE MAP
DAYTON SOUTH, OHIO

MORAINE
OHIO

figure K-3.1

SITE LOCATION MAP
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-07(005)GN-WA001 DEC 19/2006



LEGEND

- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- PARCEL BOUNDARY
- 3264 LOT NUMBER
- EDGE OF WATER

figure K-3.2

SITE PLAN
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.

38443-07(005)GN-WA002 DEC 19/2006

LEGEND

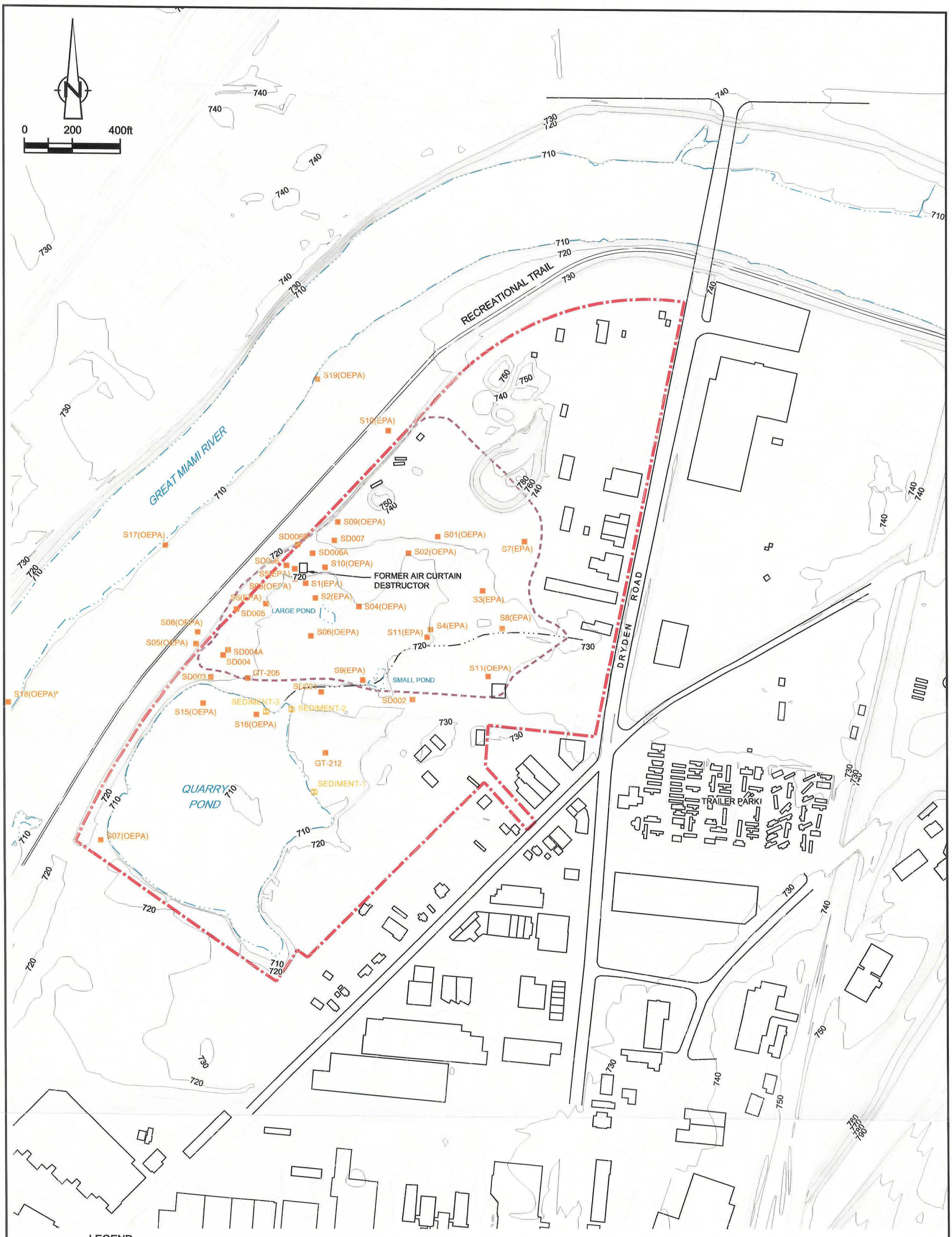
- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- 730 EXISTING GROUND CONTOUR (2 FT CONTOUR INTERVAL)
- EDGE OF WATER
- MW-206 INTERMEDIATE ZONE MONITORING WELL LOCATION
- P-211 PIEZOMETER LOCATION
- EXISTING STRUCTURE
- * APPROXIMATE LOCATION FOR MONITORING WELL MW-104

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.

figure K-3.3

EXISTING MONITORING WELL NETWORK
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-07(005)GN-WA004 DEC 19/2006



LEGEND

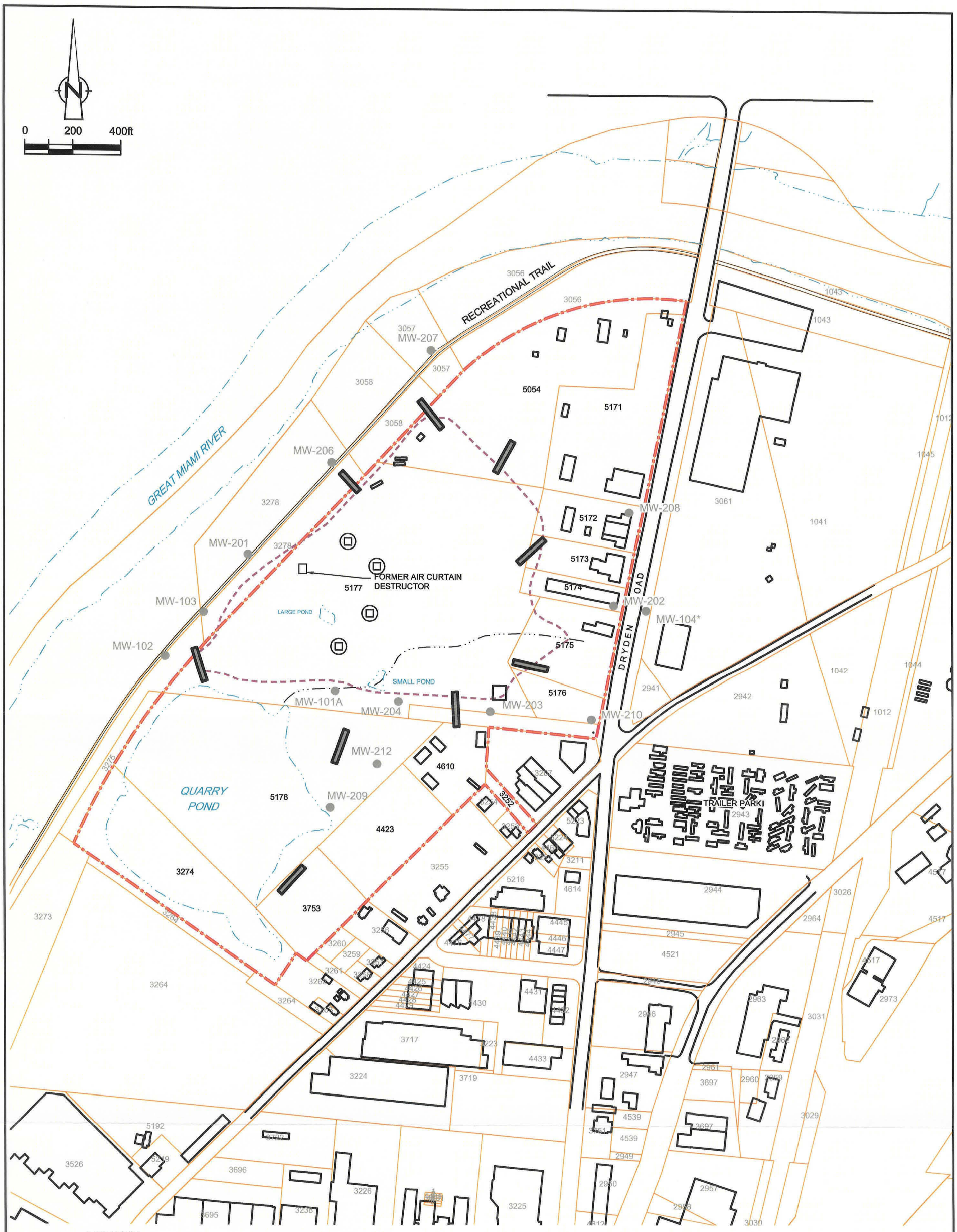
- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- 730 EXISTING GROUND CONTOUR (2 FT CONTOUR INTERVAL)
- S01 SOIL BORING LOCATION
- SEDIMENT-1 SEDIMENT SAMPLING LOCATION
- EDGE OF WATER

figure K-3.4

HISTORICAL SOIL SAMPLING AND BOREHOLE LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.

38443-07(005)GN-WA003 DEC 19/2006



LEGEND

- MW-206 ● INTERMEDIATE ZONE MONITORING WELL LOCATION
- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- PROPOSED TEST TRENCH LOCATION
- PROPOSED TEST PIT LOCATION
- EDGE OF WATER
- EXISTING STRUCTURE
- * APPROXIMATE LOCATION FOR MONITORING WELL MW-104

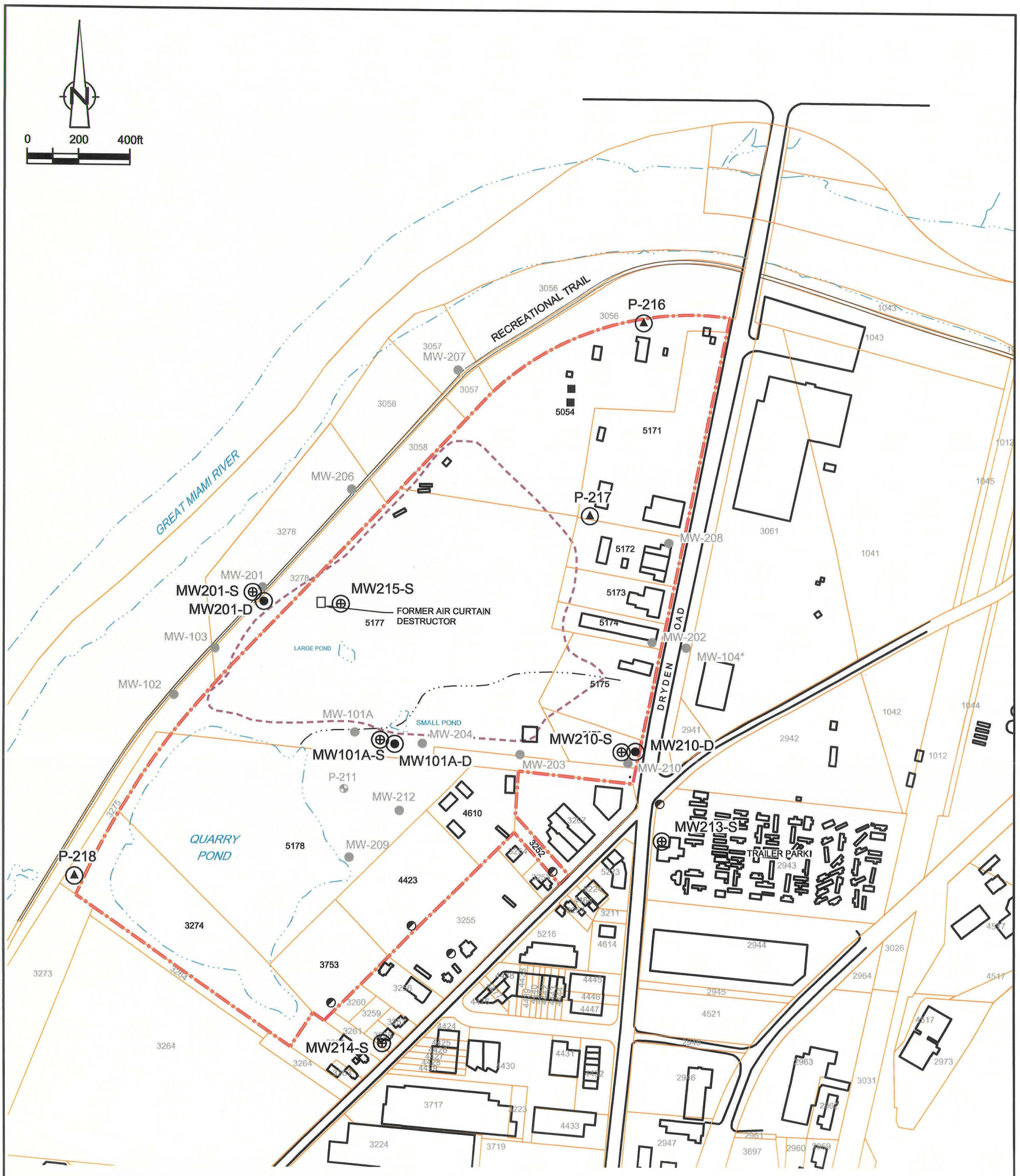
figure K-4.1

PROPOSED PHASE I TEST PIT AND TRENCH EXCAVATION LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE

Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
USGS AERIAL PHOTOGRAPH, DAYTON SOUTH, 1994.

38443-07(005)GN-WA006 JAN 05/2007



LEGEND

- MW-206 — INTERMEDIATE ZONE MONITORING WELL LOCATION
- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- EDGE OF WATER
- PROPOSED SHALLOW GROUNDWATER MONITORING WELL LOCATION
- PROPOSED DEEP GROUNDWATER MONITORING WELL LOCATION
- PROPOSED PIEZOMETER WELL LOCATION
- WATER SUPPLY WELL
- P-211 — EXISTING PIEZOMETER WELL LOCATION
- TEMPORARY WELL (GEOPROBE) LOCATION
- EXISTING STRUCTURE
- * APPROXIMATE LOCATION FOR MONITORING WELL MW-104

figure K-4.2

PROPOSED MONITORING/PIEZOMETER WELL LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE

Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
USGS AERIAL PHOTOGRAPH, DAYTON SOUTH, 1994.

38443-07(005)GN-WA007 JAN 05/2007

LEGEND

- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- 730 — EXISTING GROUND CONTOUR (2 FT CONTOUR INTERVAL)
- S01 ■ HISTORIC SOIL BORING LOCATION
- SEDIMENT-1 ⊠ HISTORIC SEDIMENT SAMPLING LOCATION
- EDGE OF WATER
- SURFACE WATER GAUGING STATION
- SEDIMENT SAMPLING LOCATION

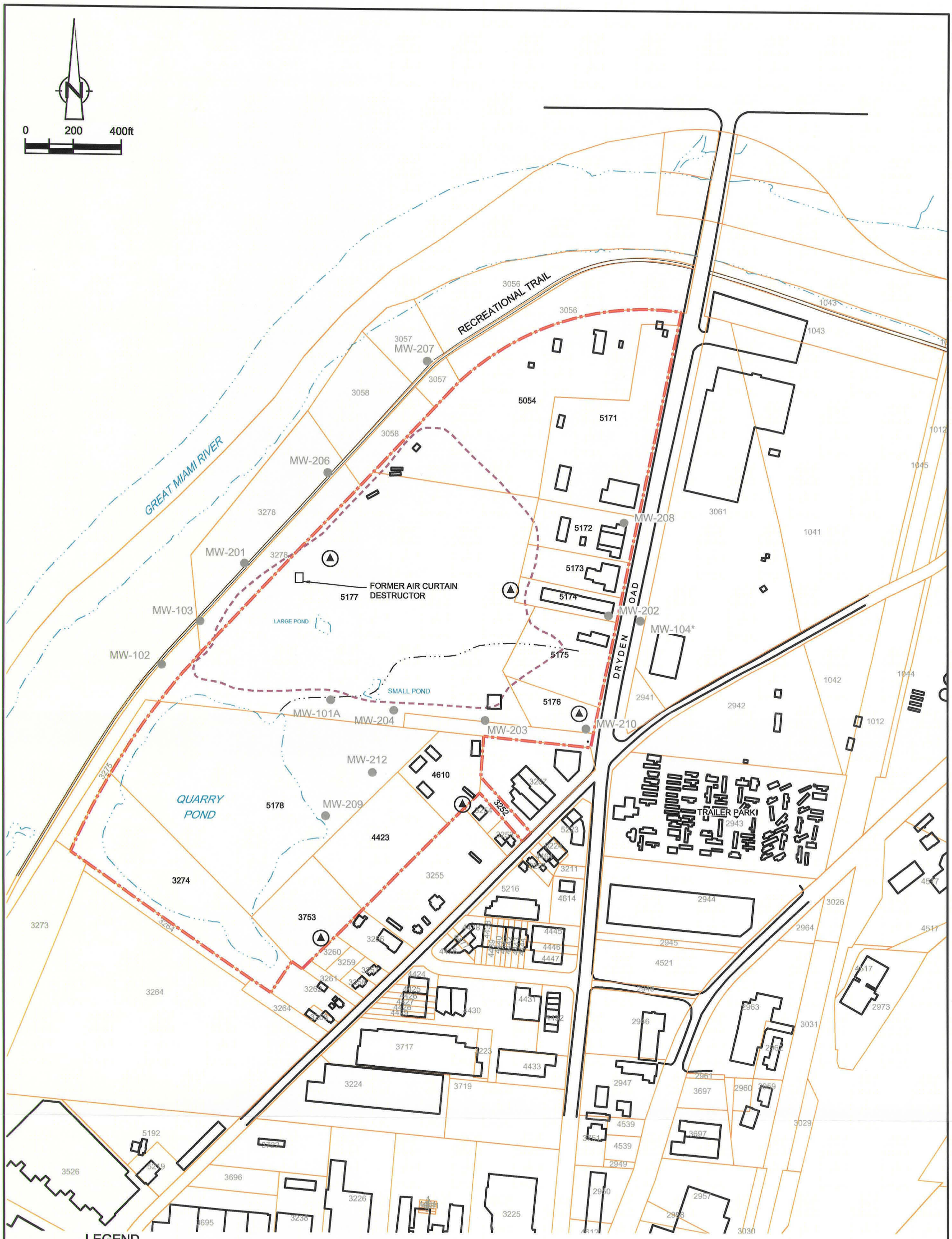
figure K-4.3

PROPOSED PHASE I SURFACE WATER AND SEDIMENT SAMPLING
SOUTH DAYTON DUMP AND LANDFILL SITE

Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.

38443-07(005)GN-WA008 JAN 05/2007



LEGEND

- MW-206 ● INTERMEDIATE ZONE MONITORING WELL LOCATION
- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- EDGE OF WATER
- PROPOSED LANDFILL GAS PROBE LOCATION
- EXISTING STRUCTURE
- * APPROXIMATE LOCATION FOR MONITORING WELL MW-104

figure K-4.4

PROPOSED PHASE I SOIL GAS PROBE LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE

Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
USGS AERIAL PHOTOGRAPH, DAYTON SOUTH, 1994.

38443-07(005)GN-WA009 JAN 05/2007

TABLE K.3.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Task/Event	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Sample Locations*	*Investigative Samples*	*Quality Control Samples*[1] *Field Blanks*[2]	*Field Duplicates*	*MS/MSD LCS/LCD*[3]	*Total*[4]
Groundwater Investigation									
Vertical Aquifer Sampling (VAS) (5 foot intervals - maximum 100 foot depths)	Water	pH,/Temperature, Conductivity, DO, Turbidity, ORP	TCL VOCs, Total Arsenic, Total Lead	25	400[5]	63	32	32	495
			TCL SVOCs	25	100	10	5	5	115
Existing Monitoring Wells	Water	pH,/Temperature, Conductivity, DO, Turbidity, ORP, Iron (II)	TCL VOC, TCL SVOC, Dissolved Arsenic, Dissolved Lead	13	26	2	2	2	30
Additional Monitoring Wells	Water	pH,/Temperature, Conductivity, DO, Turbidity, ORP	TCL VOCs, TCL SVOCs, TAL Inorganics[6], MNA[7] parameters as appropriate.	TBD	TBD	1/10	1/10	1/20	TBD
Landfill Seep Investigation									
Seep Characterization	Water		TCL VOCs, TCL SVOCs, TCL Pesticides, PCBs, TAL Inorganics[6]	TBD	TBD	1/10	1/10	1/20	TBD
	Soil		TCL VOCs, TCL SVOCs, TCL Pesticides, PCBs, TAL Inorganics[6], Asbestos	TBD	TBD	1/10	1/10	1/20	TBD
Landfill Gas Investigation									
Soil Gas Sampling (Two Rounds)									
- Round 1	Air	Gas Pressure, Methane, Oxygen LEL Screen	Select VOC	18	18	1[8]	1	1	20
- Round 2	Air	Gas Pressure, Methane, Oxygen LEL Screen	Select VOC	18	18	1[8]	1	1	20
Test Pit/Test Trench Investigation									
Test Pit Sampling	Solid	PID Screen	TCL VOCs, TCL SVOCs, TCL Pesticides, PCBs, RCRA Herbicides, TAL Inorganics[6]	6	6	1	1	1	8
Test Trench Sampling	Solid	PID Screen	TCL VOCs, TCL SVOCs, TCL Pesticides, PCBs, RCRA Herbicides, TAL Inorganics[6]	23	46	4	2	2	52
Ash Fill Materials	Solid	Visual	Dioxins & Furans	TBD	TBD	1/10	1/10	1/20	TBD
Potential Asbestos Containing Materials	Solid	Visual	Asbestos	TBD	TBD				TBD
Leachate Sampling	Solid	PID Screen	TCL VOCs, TCL SVOCs, TCL Pesticides, PCBs, RCRA Herbicides, TAL Inorganics[6]	TBD	TBD	1/10	1/10	1/20	TBD

TABLE K.3.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Task/Event	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Sample Locations*	*Investigative Samples*	*Quality Control Samples*[1] *Field Blanks*[2]	*Field Duplicates*	*MS/MSD LCS/LCD*[3]	*Total*[4]
Drum and Waste Characterization	Solid / Water	PID Screen	TCLP VOCs, TCLP SVOCs, TCLP Herbicides, TCLP Pesticides, TCLP Metals, PCBs, Ignitibility, Total Cyanide, Total Sulfide, Corrosivity	TBD	TBD	--	--	--	TBD

Notes:

1 Quality control samples will include laboratory supplied trip blank samples for volatile sample analysis with each shipping cooler of aqueous investigative samples.
2 Field blank samples consisting of equipment rinsate blanks will not be collected when dedicated or disposable sampling equipment is employed.
3 Matrix spike/matrix spike duplicate (MS/MSD) or laboratory control sample/laboratory control duplicate (LCS/LCD) in the case of air samples are required for each batch of 20 samples submitted.
4 The total quantity does not include MS/MSD (LCS/LCD) samples and is dependent on the actual quantity of field quality control samples collected.
5 Number shown is maximum possible number. Total number of samples will be dependent on depth to groundwater at each location.
6 TAL Inorganics include the 23 metals and total cyanide.
7 MNA - Monitored Natural Attenuation Parameters include alkalinity, chloride, dissolved organic carbon, hardness, nitrate, nitrite, sulfate, sulfite, select metals (Ca, Mg, Mn), and dissolved gases.
8 Soil gas sampling will include one ambient air sample per event.

TCL - Target Compound List
VOC - Volatile Organic Compounds
SVOC - Semi-volatile Organic Compounds
TAL - Target Analyte List
PCB - Polychlorinated Biphenyls
TCLP - Toxic Characteristics Leachate Procedure
DO - Dissolve Oxygen
ORP - Oxygen Reduction Potential

TABLE K.3.2

INVESTIGATIVE PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1]		*Method Detection Limits (MDL)* [2]	
Compound	*Water (µg/L)*	*Solid (µg/kg)*	*Water (µg/L)*	*Solid (µg/kg)*
TCL Volatile Organic Compounds (VOC)				
Acetone	10	20	1.1	6.3
Benzene	1	5	0.13	0.23
Bromodichloromethane	1	5	0.15	0.28
Bromoform	1	5	0.64	0.33
Bromomethane	1	5	0.41	0.54
2-Butanone	10	20	0.57	1.4
Carbon disulfide	1	5	0.13	0.44
Carbon tetrachloride	1	5	0.13	0.37
Chlorobenzene	1	5	0.15	0.33
Dibromochloromethane	1	5	0.18	0.55
Chloroethane	1	5	0.29	0.86
Chloroform	1	5	0.16	0.29
Chloromethane	1	5	0.3	0.41
Cyclohexane	1	10	0.12	0.33
1,2-Dibromo-3-chloropropane	2	10	0.67	1.3
1,2-Dibromoethane	1	5	0.24	0.5
1,2-Dichlorobenzene	1	5	0.13	0.36
1,3-Dichlorobenzene	1	5	0.14	0.35
1,4-Dichlorobenzene	1	5	0.13	0.66
Dichlorodifluoromethane	1	5	0.31	0.5
1,1-Dichloroethane	1	5	0.15	0.36
1,2-Dichloroethane	1	5	0.22	0.34
cis-1,2-Dichloroethene	1	5	0.17	0.36
trans-1,2-Dichloroethene	1	5	0.19	0.41
1,1-Dichloroethene	1	5	0.19	0.52
1,2-Dichloropropane	1	5	0.18	0.69
cis-1,3-Dichloropropene	1	5	0.14	0.34
trans-1,3-Dichloropropene	1	5	0.19	0.54
Ethylbenzene	1	5	0.17	0.26
2-Hexanone	10	20	0.41	0.63
Isopropylbenzene	1	5	0.13	0.16
Methyl acetate	10	10	0.38	1.4
Methylcyclohexane	1	10	0.13	0.31
Methylene chloride	1	5	0.33	0.67
4-Methyl-2-pentanone	10	20	0.32	0.54
Methyl tert-butyl ether	5	20	0.17	0.43
Styrene	1	5	0.11	0.15
1,1,2,2-Tetrachloroethane	1	5	0.18	0.34
Tetrachloroethene	1	5	0.29	0.52
Toluene	1	5	0.13	0.27
1,2,4-Trichlorobenzene	1	5	0.15	0.27
1,1,1-Trichloroethane	1	5	0.22	0.56

TABLE K.3.2

INVESTIGATIVE PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1]		*Method Detection Limits (MDL)* [2]	
Compound	*Water (µg/L)*	*Solid (µg/kg)*	*Water (µg/L)*	*Solid (µg/kg)*
TCL VOC (continued)				
1,1,2-Trichloroethane	1	5	0.27	0.39
Trichloroethene	1	5	0.17	0.42
Trichlorofluoromethane	1	5	0.21	0.34
1,1,2-Trichloro-1,2,2-trifluoroethane	1	5	0.28	1.3
Vinyl chloride	1	5	0.22	0.39
Xylenes (total)	2	10	0.28	0.67
TCL Semi-Volatile Organic Compounds (SVOC)				
Acenaphthene	0.2	6.67	0.054	1.3
Acenaphthylene	0.2	6.67	0.054	1.2
Acetophenone	1	100	0.55	26
Anthracene	0.2	6.67	0.054	1.3
Atrazine	1	200	0.65	21
Benzaldehyde	1	100	0.75	21
Benzo(a)anthracene	0.2	6.67	0.052	0.95
Benzo(b)fluoranthene	0.2	6.67	0.049	1.2
Benzo(k)fluoranthene	0.2	6.67	0.049	1.7
Benzo(ghi)perylene	0.2	6.67	0.053	1.3
Benzo(a)pyrene	0.2	6.67	0.048	1.3
1,1'-Biphenyl	1	50	0.55	23
bis(2-Chloroethoxy)methane	1	100	0.49	22
bis(2-Chloroethyl) ether	1	100	0.088	2
bis(2-Ethylhexyl) phthalate	2	50	0.88	18
4-Bromophenyl phenyl ether	2	50	0.52	21
Butyl benzyl phthalate	1	50	0.51	19
Caprolactam	5	330	0.61	37
Carbazole	1	50	0.54	19
4-Chloroaniline	2	150	0.56	17
4-Chloro-3-methylphenol	2	150	0.41	21
2-Chloronaphthalene	1	50	0.62	22
2-Chlorophenol	1	50	0.039	26
4-Chlorophenyl phenyl ether	2	50	0.55	24
Chrysene	0.2	6.67	0.048	0.9
Dibenz(a,h)anthracene	0.2	6.67	0.039	1.3
Dibenzofuran	1	50	0.54	20
Di-n-butyl phthalate	1	50	0.61	19
3,3'-Dichlorobenzidine	5	100	0.48	18
2,4-Dichlorophenol	2	150	1.1	20
Diethyl phthalate	1	50	0.63	19
2,4-Dimethylphenol	2	150	0.56	20
Dimethyl phthalate	1	50	0.44	21
4,6-Dinitro-2-methylphenol	5	150	0.27	13

TABLE K.3.2

INVESTIGATIVE PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1]		*Method Detection Limits (MDL)* [2]	
Compound	*Water (µg/L)*	*Solid (µg/kg)*	*Water (µg/L)*	*Solid (µg/kg)*
TCL SVOC (continued)				
2,4-Dinitrophenol	5	330	3.5	83
2,4-Dinitrotoluene	5	200	0.4	18
2,6-Dinitrotoluene	5	200	0.47	21
Di-n-octyl phthalate	1	50	0.39	18
Fluoranthene	0.2	6.67	0.036	1.2
Fluorene	0.2	6.67	0.043	1.2
Hexachlorobenzene	0.2	6.67	0.065	2.1
Hexachlorobutadiene	1	50	0.51	26
Hexachlorocyclopentadiene	10	330	0.74	16
Hexachloroethane	1	50	0.58	28
Indeno(1,2,3-cd)pyrene	0.2	6.67	0.065	1.5
Isophorone	1	50	0.5	21
2-Methylnaphthalene	0.2	6.67	0.061	1.5
2-Methylphenol	1	200	0.56	28
4-Methylphenol	1	200	0.64	22
Naphthalene	0.2	6.67	0.069	1.6
2-Nitroaniline	2	200	0.43	22
3-Nitroaniline	2	200	0.67	16
4-Nitroaniline	2	200	0.47	26
Nitrobenzene	1	100	0.053	2.2
2-Nitrophenol	2	50	1.3	19
4-Nitrophenol	5	330	0.63	110
N-Nitrosodiphenylamine	1	50	0.46	21
N-Nitrosodi-n-propylamine	1	50	0.53	23
2,2'-oxybis(1-Chloropropane)	1	100	0.52	26
Pentachlorophenol	5	150	0.48	82
Phenanthrene	0.2	6.67	0.087	2
Phenol	1	50	0.96	25
Pyrene	0.2	6.67	0.048	1.1
2,4,5-Trichlorophenol	5	150	0.96	25
2,4,6-Trichlorophenol	5	150	1.4	21
Polychlorinated Biphenyls (PCB) as Aroclors				
Aroclor-1016 (PCB-1016)	0.2	33	0.044	21
Aroclor-1221 (PCB-1221)	0.2	33	0.045	16
Aroclor-1232 (PCB-1232)	0.2	33	0.073	14
Aroclor-1242 (PCB-1242)	0.2	33	0.06	13
Aroclor-1248 (PCB-1248)	0.2	33	0.061	17
Aroclor-1254 (PCB-1254)	0.2	33	0.032	17
Aroclor-1260 (PCB-1260)	0.2	33	0.038	17
Herbicides				
2,4-Dichlorophenoxyacetic acid (2,4-D)	--	80	--	36
2,4,5-Trichlorophenoxyacetic acid (2,4,5-TP)	--	20	--	2.2

TABLE K.3.2

INVESTIGATIVE PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1]		*Method Detection Limits (MDL)* [2]	
Compound	*Water (µg/L)*	*Solid (µg/kg)*	*Water (µg/L)*	*Solid (µg/kg)*
TCL Pesticides				
Aldrin	0.05	1.7	0.0082	1.2
alpha-BHC	0.05	1.7	0.007	0.73
beta-BHC	0.05	1.7	0.0084	1.1
delta-BHC	0.05	1.7	0.0087	1.2
gamma-BHC (Lindane)	0.05	1.7	0.0064	0.74
alpha-Chlordane	0.05	1.7	0.014	0.94
gamma-Chlordane	0.05	1.7	0.012	0.42
4,4'-DDD	0.05	1.7	0.0096	0.62
4,4'-DDE	0.05	1.7	0.0097	0.39
4,4'-DDT	0.05	1.7	0.016	0.63
Dieldrin	0.05	1.7	0.0075	0.47
Endosulfan I	0.05	1.7	0.013	0.52
Endosulfan II	0.05	1.7	0.012	0.82
Endosulfan sulfate	0.05	1.7	0.011	0.87
Endrin	0.05	1.7	0.011	0.5
Endrin aldehyde	0.05	1.7	0.011	1
Endrin ketone	0.05	1.7	0.0078	0.63
Heptachlor	0.05	1.7	0.008	1.1
Heptachlor epoxide	0.05	1.7	0.0071	0.8
Methoxychlor	0.1	3.3	0.032	1.5
Toxaphene	2	67	0.32	19

Parameter	*Targeted Quantitation Limit (TQL)* [3]		*Method Detection Limits (MDL)* [4]	
Compound	*Water (pg/L)*	*Soil/Sediment (ng/kg)*	*Water (pg/L)*	*Soil/Sediment (ng/kg)*
Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/PCDF)				
2,3,7,8 - Tetrachlorodibenzo-p-dioxin (TCDD)	10	1.0	- -4	- -4
2,3,7, 8 - Tetrachlorodibenzofuran (TCDF)	10	1.0	- -	- -
1,2,3,7,8 - Pentachlorodibenzo-p-dioxin (PeCDD)	50	5.0	- -	- -
1,2,3,7,8 - Pentachlorodibenzofuran (PeCDF)	50	5.0	- -	- -
2,3,4,7,8 - Pentachlorodibenzofuran (PeCDF)	50	5.0	- -	- -
1,2,3,4,7,8 - Hexachlorodibenzo-p-dioxin (HxCDD)	50	5.0	- -	- -
1,2,3,6,7,8 - Hexachlorodibenzo-p-dioxin (HxCDD)	50	5.0	- -	- -
1,2,3,7,8,9 - Hexachlorodibenzo-p-dioxin (HxCDD)	50	5.0	- -	- -
1,2,3,4,7,8 - Hexachlorodibenzofuran (HxCDF)	50	5.0	- -	- -
1,2,3,6,7,8 - Hexachlorodibenzofuran (HxCDF)	50	5.0	- -	- -
1,2,3,7,8,9 - Hexachlorodibenzofuran (HxCDF)	50	5.0	- -	- -
2,3,4,6,7,8 - Hexachlorodibenzofuran (HxCDF)	50	5.0	- -	- -
1,2,3,4,6,7,8 - Heptachlorodibenzo-p-dioxin (HpCDD)	50	5.0	- -	- -
1,2,3,4,6,7,8 - Heptachlorodibenzofuran (HpCDF)	50	5.0	- -	- -
1,2,3,4,7,8,9 - Heptachlorodibenzofuran (HpCDF)	50	5.0	- -	- -
Octachlorodibenzo-p-dioxin (OCDD)	100	10	- -	- -
Octachlorodibenzofuran (OCDF)	100	10	- -	- -

TABLE K.3.2

INVESTIGATIVE PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1]		*Method Detection Limits (MDL)* [2]	
Compound	*Water (µg/L)*	*Solid (mg/kg)*	*Water (µg/L)*	*Solid (mg/kg)*
Target Analyte List (TAL) Metals				
Aluminum	200	20	97	9.6
Antimony	2	6	0.13	0.39
Arsenic	5	0.5	0.4	0.062
Barium	200	20	0.67	0.071
Beryllium	5	0.5	0.46	0.043
Cadmium	1	0.5	0.13	0.036
Calcium	5000	500	130	16
Chromium Total	10	1	2.2	0.2
Cobalt	50	5	1.7	0.16
Copper	25	2.5	4.5	0.74
Iron	100	10	81	4.9
Lead	1	10	0.18	0.19
Magnesium	5000	500	34	5.1
Manganese	15	1.5	0.41	0.074
Mercury	0.2	0.1	0.12	0.015
Nickel	40	4	3.2	0.27
Potassium	5000	500	72	6.2
Selenium	5	25	1.2	0.45
Silver	1	1	0.08	0.1
Sodium	5000	500	590	66
Thallium	1	0.1	0.14	0.013
Vanadium	50	5	0.64	0.12
Zinc	20	2	2.3	1
Cyanide, total	10	0.5	5.0	0.10

Parameter	*Targeted Quantitation Limit (TQL)* [1] *Soil/Sediment (%)*
Asbestos	***<0.25***

Notes:

[1] - Please note that these are targeted quantitation limits and are presented for guidance only. Actual quantitation limits are highly matrix dependent and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes.

[2] - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory

[3] - MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes (where ND = 0).
The EPA 1998 toxic equivalency factor or TEF will be used to determine the toxic equivalency (TEQ) concentration for each congener.

[4] - Sample specific MDLs or estimated detection limits (EDL) are determined for each sample based on signal-to-noise ratios at the analyte retention time.

TABLE K.3.3

MONITORED NATURAL ATTENUATION PARAMETER LISTS AND TARGETED QUANTITATION LIMITS REMEDIAL INVESTIGATION/FEASIBILITY STUDY SOUTH DAYTON DUMP AND LANDFILL MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1]	*Method Detection Limits (MDL)* [2]
Compound	*Water (mg/L)*	*Water (mg/L)*
Monitored Natural Attenuation (MNA) Inorganics		
Alkalinity	5	1.9
Chloride	1	0.1
Dissolved Organic Carbon (1)	1	0.24
Hardness, total	33	33
Hardness, carbonate	33	33
Nitrate	0.1	0.023
Nitrite	0.1	0.012
Sulfate	1	0.12
Sulfide	1	0.86
MNA Metals		
Calcium	1	0.022
Magnesium	1	0.017
Manganese (II) (Mn^{2+}) (1)	0.001	0.00083

	Targeted Quantitation Limits (EQL) [1]	*Method Detection Limits (MDL)* [2]
Compound	*Water (µg/L)*	*Water (µg/L)*
MNA Dissolved Gases		
Methane	0.5	0.17
Ethane	0.5	0.27
Ethene	0.5	0.24

Notes:

[1] - Please note that these are targeted quantitation limits and are presented for guidance only. Actual quantitation limits are highly matrix dependent and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes.

[2] - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.

TABLE K.3.4

SOIL GAS PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1]	*Method Detection Limits (MDL)* [2]	*PRG*
Compound	*Air* ($\mu g/M^3$)	*Air* ($\mu g/M^3$)	*Ambient Air* ($\mu g/M^3$)
Select Volatile Organic Compounds (VOC)			
Acetone	24	5.9	3300
Benzene	0.96	0.64	0.21
Bromodichloromethane	2.0	1.6	0.11
Bromoform	4.1	2.1	1.7
Bromomethane	16	7.8	5.2
2-Butanone	29	5.9	5100
Carbon disulfide	31	6.2	730
Carbon tetrachloride	1.9	1.3	13
Chlorobenzene	1.4	0.92	62
Chloroethane	1.1	1.0	2.3
Chloroform	1.5	0.97	830
Chloromethane	1.6	0.82	95
Cyclohexane	1.7	1.4	6300
Dibromochloromethane	3.4	1	0.08
1,2-Dibromo-3-chloropropane	9.6	3.9	0.21
1,2-Dibromoethane	3.1	1.5	0.0034
1,2-Dichlorobenzene	2.4	1.2	21
1,3-Dichlorobenzene	2.4	1.2	11
1,4-Dichlorobenzene	2.4	1.2	31
Dichlorodifluoromethane	1.5	0.99	21
1,1-Dichloroethane	1.2	0.81	52
1,2-Dichloroethane	8.1	4.0	74
1,1-Dichloroethene	7.9	4.0	21
cis-1,2-Dichloroethene	7.9	3.2	37
trans-1,2-Dichloroethene	7.9	4.0	73
1,2-Dichloropropane	14	6.9	0.099
cis-1,3-Dichloropropene	1.8	0.91	0.48
trans-1,3-Dichloropropene	1.8	0.91	0.48
Ethylbenzene	1.3	0.87	1100
2-Hexanone	2.0	1.6	NA
Isopropylbenzene	2.5	2.0	400
Methylene chloride	1	0.69	4.1
4-Methyl-2-pentanone	41	8.2	3100
Methyl tert-butyl ether	7.2	3.6	7.4
Styrene	1.7	0.85	1100
1,1,2,2-Tetrachloroethane	14	6.8	0.033
Tetrachloroethene	2.7	1.4	0.32
Toluene	7.5	3.8	400
1,2,4-Trichlorobenzene	37	18	3.7
1,1,1-Trichloroethane	1.6	1.1	2300

TABLE K.3.4

SOIL GAS PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1]	*Method Detection Limits (MDL)* [2]	*Method Detection Limits (MDL)* [2]
Compound	*Air* ($\mu g/M^3$)	*Air* ($\mu g/M^3$)	*Air* ($\mu g/M^3$)
Select VOC (continued)			
1,1,2-Trichloroethane	1.6	1.1	N/A
Trichloroethene	2.1	1.1	N/A
Trichlorofluoromethane	11	5.6	N/A
1,1,2-Trichloro-1,2,2-trifluoroethane	3.8	3.1	N/A
Vinyl chloride	7.6	3.8	N/A
Xylenes (total)	17	4.3	N/A

Notes:

[1] - Please note that these are targeted quantitation limits and are presented for guidance only. Actual quantitation limits are highly matrix dependent and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes.

[2] - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.

TABLE K.3.5

WASTE CHARACTERIZATION PARAMETER LIST AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Paramater	*Targeted Quantitation Limits (EQL)* [1]	*Method Detection Limits (MDL)* [2]
Compound	*Waste Leachate (mg/L)*	*Waste Leachate (mg/L)*
Toxic Characteristic Leachate Procedure (TCLP) VOC		
Benzene	0.025	0.00013
2-Butanone	0.25	0.00057
Carbon tetrachloride	0.025	0.00013
Chlorobenzene	0.025	0.00015
Chloroform	0.025	0.00016
1,2-Dichloroethane	0.025	0.00022
1,1-Dichloroethene	0.07	0.00019
Tetrachloroethene	0.07	0.00029
Trichloroethene	0.05	0.00017
Vinyl chloride	0.025	0.00022
TCLP SVOC		
m-Cresols & p-Cresol	0.04	0.00075
o-Cresol	0.004	0.00056
1,4-Dichlorobenzene	0.004	0.00052
2,4-Dinitrotoluene	0.02	0.0004
Hexachlorobenzene	0.02	0.000065
Hexachlorobutadiene	0.02	0.00051
Hexachloroethane	0.02	0.00058
Nitrobenzene	0.004	0.000053
Pentachlorophenol	0.04	0.00048
Pyridine	0.02	0.00078
2,4,5-Trichlorophenol	0.02	0.00096
2,4,6-Trichlorophenol	0.02	0.0014
TCLP Pesticides		
Chlordane	0.005	0.033
Endrin	0.0005	0.011
Heptachlor	0.0005	0.008
Heptachlor epoxide	0.0005	0.0071
gamma-BHC (Lindane)	0.0005	0.0064
Methoxychlor	0.001	0.032
Toxaphene	0.02	0.32
TCLP Herbicides		
2,4-Dichlorophenoxyacetic acid (2,4-D)	0.5	1.5
2,4,5-Trichlorophenoxyacetic acid (2,4,5-TP)	0.1	0.16

TABLE K.3.5

WASTE CHARACTERIZATION PARAMETER LIST AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Paramater	*Targeted Quantitation Limits (EQL)* [1]	*Method Detection Limits (MDL)* [2]
Compound	*Waste Leachate (mg/L)*	*Waste Leachate (mg/L)*
TCLP Metals		
Arsenic	0.5	0.0032
Barium	10	0.0067
Cadmium	0.1	0.00066
Chromium	0.5	0.0022
Lead	0.5	0.0019
Mercury	0.002	0.00012
Selenium	0.25	0.0041
Silver	0.5	0.0022

	Targeted Quantitation Limits (EQL) [1]	*Method Detection Limits (MDL)* [2]
	Waste (mg/kg)	*Waste (mg/kg)*
Waste Characteristics		
Ignitablilty (flashpoint)	---	NA
Corrosivity (pH)	---	NA

	Targeted Quantitation Limit (TQL) [1]		*Method Detection Limits (MDL)* [2]	
	Water (mg/L)	*Solid (mg/kg)*	*Water (mg/L)*	*Solid (mg/kg)*
Cyanide, Total	0.01	0.5	0.005	0.1
Sulfide, Total	1	100	0.86	11

Notes:

[1] - Please note that these are estimated quantitation limits and are presented for guidance only. Actual quantitation limits are highly matrix dependent and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes.

[2] - Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.

TABLE K.4.1

SOIL AND GROUNDWATER CLEANUP CRITERIA
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Soil PRGs Residential*	*Soil PRGs Industrial*	*PRG Tap Water*	*PRG Ambient Air*
Compound	*(µg/kg)*	*(µg/kg)*	*(µg/L)*	*(µg/M³)*
TCL Volatile Organic Compounds (VOC)				
Acetone	14000000	54000000	5500	3300
Benzene	640	1400	0.35	0.21
Bromodichloromethane	820	1800	0.18	0.11
Bromoform	62000	220000	8.5	1.7
Bromomethane (Methyl Bromide)	3900	13000	8.7	5.2
2-Butanone (Methyl Ethyl Ketone)	22000000	110000000	7000	5100
Carbon disulfide	360000	720000	1000	730
Carbon tetrachloride	250	550	0.17	13
Chlorobenzene	150000	530000	110	62
Chloroethane	3000	6500	4.6	2.3
Chloroform (Trichloromethane)	220	470	0.17	830
Chloromethane (Methyl Chloride)	47000	160000	160	95
Cyclohexane	140000	140000	10000	6300
Dibromochloromethane	1100	2600	0.13	0.08
1,2-Dibromo-3-chloropropane (DBCP)	460	2000	0.048	0.21
1,2-Dibromoethane (Ethylene Dibromide)	32	73	0.0056	0.0034
1,2-Dichlorobenzene	600000	600000	370	21
1,3-Dichlorobenzene	530000	600000	180	11
1,4-Dichlorobenzene	3400	7900	0.5	31
Dichlorodifluoromethane (CFC-12)	94000	310000	390	21
1,1-Dichloroethane	510000	1700000	810	52
1,2-Dichloroethane	280	600	0.12	74
1,1-Dichloroethene	120000	410000	340	21
cis-1,2-Dichloroethene	43000	150000	61	37
trans-1,2-Dichloroethene	69000	230000	120	73
1,2-Dichloropropane	340	740	0.16	0.099
cis-1,3-Dichloropropene	780	1800	0.4	0.48
trans-1,3-Dichloropropene	780	1800	0.4	0.48
Ethylbenzene	400000	400000	1300	1100
2-Hexanone	NA	NA	NA	NA
Isopropylbenzene	160000	520000	660	400
Methyl acetate	22000000	92000000	6100	NA
Methylene chloride	9100	21000	4.3	4.1
Methyl cyclohexane	2600000	8700000	5200	NA
4-Methyl-2-Pentanone (Methyl Isobutyl Ketone	5300000	47000000	2000	3100
Methyl Tert Butyl Ether	32000	70000	11	7.4
Styrene	1700000	1700000	1600	1100
1,1,2,2-Tetrachloroethane	410	930	0.055	0.033
Tetrachloroethene	480	1300	0.1	0.32
Toluene	520000	520000	720	400
1,2,4-Trichlorobenzene	62000	220000	7.2	3.7
1,1,1-Trichloroethane	1200000	1200000	3200	2300
1,1,2-Trichloroethane	730	1600	0.2	0.12

TABLE K.4.1

SOIL AND GROUNDWATER CLEANUP CRITERIA
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter / *Compound*	*Soil PRGs Residential* (*µg/kg*)	*Soil PRGs Industrial* (*µg/kg*)	*PRG Tap Water* (*µg/L*)	*PRG Ambient Air* (*µg/M³*)
TCL VOC (Continued)				
Trichloroethene	53	110	0.028	0.017
Trichlorofluoromethane (CFC-11)	390000	2000000	1300	730
Trifluorotrichloroethane (Freon 113)	5600000	5600000	59000	31000
Vinyl chloride	79	750	0.02	0.11
Xylene (total)	270000	420000	210	110
TCL Semi-Volatile Organic Compounds (SVOC)				
Acenaphthene	NA	NA	NA	NA
Acenaphthylene	NA	NA	NA	NA
Acetophenone	NA	NA	NA	NA
Anthracene	22000000	100000000	1800	NA
Atrazine	2200	7800	0.3	NA
Benzaldehyde	6100000	62000000	3600	NA
Benzo(a)anthracene	620	2100	0.092	NA
Benzo(a)pyrene	62	210	0.0092	NA
Benzo(b)fluoranthene	620	2100	0.092	NA
Benzo(g,h,i)perylene	NA	NA	NA	NA
Benzo(k)fluoranthene	6200	21000	0.92	NA
Biphenyl	3000000	23000000	300	NA
4-Bromophenyl phenyl ether	NA	NA	NA	NA
Butyl benzylphthalate	12000000	100000000	7300	NA
Di-n-butylphthalate	6100000	62000000	3600	NA
Caprolactam	31000000	100000000	18000	NA
Carbazole	24000	86000	3.4	NA
4-Chloroaniline	240000	2500000	150	NA
bis(2-Chloroethoxy)methane	NA	NA	NA	NA
bis(2-Chloroethyl)ether	220	580	0.01	NA
2,2'-oxybis(1-Chloropropane) (bis(2-chloroisop	2900	7400	0.27	NA
4-Chloro-3-methylphenol	NA	NA	NA	NA
2-Chloronaphthalene	4900000	23000000	490	NA
2-Chlorophenol	63000	240000	30	NA
4-Chlorophenyl phenyl ether	NA	NA	NA	NA
Chrysene	62000	210000	9.2	NA
Dibenz(a,h)anthracene	62	210	0.0092	NA
Dibenzofuran	150000	1600000	12	NA
3,3'-Dichlorobenzidine	1100	3800	0.15	NA
2,4-Dichlorophenol	180000	1800000	110	NA
Diethyl phthalate	49000000	100000000	29000	NA
2,4-Dimethylphenol	1200000	12000000	730	NA
Dimethyl phthalate	100000000	100000000	360000	NA
4,6-Dinitro-2-methylphenol	6100	62000	3.6	NA
2,4-Dinitrophenol	120000	1200000	73	NA

TABLE K.4.1

SOIL AND GROUNDWATER CLEANUP CRITERIA
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter / *Compound*	*Soil PRGs Residential* (*µg/kg*)	*Soil PRGs Industrial* (*µg/kg*)	*PRG Tap Water* (*µg/L*)	*PRG Ambient Air* (*µg/M³*)
TCL SVOC (Continued)				
2,4-Dinitrotoluene	120000	1200000	73	NA
2,6-Dinitrotoluene	61000	620000	36	NA
bis(2-Ethylhexyl)phthalate	35000	120000	4.8	NA
Fluoranthene	2300000	22000000	1500	NA
Fluorene	2700000	26000000	240	NA
Hexachlorobenzene	300	1100	0.042	NA
Hexachlorobutadiene	6200	22000	0.86	NA
Hexachlorocyclopentadiene	370000	3700	220	NA
Hexachloroethane	35000	120000	4.8	NA
Indeno(1,2,3-cd)pyrene	620	2100	0.092	NA
Isophorone	510000	1800000	71	NA
2-Methylnaphthalene	NA	NA	NA	NA
2-Methylphenol	3100000	31000000	1800	NA
4-Methylphenol	310000	3100000	180	NA
Naphthalene	56000	190000	6.2	NA
2-Nitroaniline	180000	1800000	110	NA
3-Nitroaniline	18000	82000	3.2	NA
4-Nitroaniline	23000	82000	3.2	NA
Nitrobenzene	20000	100000	3.4	NA
2-Nitrophenol	NA	NA	NA	NA
4-Nitrophenol	490000	7000000	290	NA
N-Nitrosodi-n-propylamine	69	250	0.0096	NA
N-Nitrosodiphenylamine	99000	350000	14	NA
Di-n-octyl phthalate	2400000	25000000	1500	NA
Pentachlorophenol	3000	9000	0.56	NA
Phenanthrene	NA	NA	NA	NA
Phenol	18000000	100000000	11000	NA
Pyrene	2300000	29000000	180	NA
2,4,5-Trichlorophenol	6100000	62000000	3600	NA
2,4,6-Trichlorophenol	6100	62000	3.6	NA
Polychlorinated Biphenyls (PCB) as Aroclors				
Aroclor-1016 (PCB-1016)	3900	21000	0.96	NA
Aroclor-1221 (PCB-1221)	220	740	0.034	NA
Aroclor-1232 (PCB-1232)	220	740	0.034	NA
Aroclor-1242 (PCB-1242)	220	740	0.034	NA
Aroclor-1248 (PCB-1248)	220	740	0.034	NA
Aroclor-1254 (PCB-1254)	220	740	0.034	NA
Aroclor-1260 (PCB-1260)	220	740	0.034	NA
Herbicides				
2,4-Dichlorophenoxyacetic acid (2,4-D)	690000	7700000	360	NA
2,4,5-Trichlorophenoxyacetic acid (2,4,5-TP)	610000	6200000	36	NA

TABLE K.4.1

SOIL AND GROUNDWATER CLEANUP CRITERIA
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Compound*	*Soil PRGs Residential* (*μg/kg*)	*Soil PRGs Industrial* (*μg/kg*)	*PRG Tap Water* (*μg/L*)	*PRG Ambient Air* (*μg/M*^{3})
Pesticides					
	Aldrin	29	100	0.004	NA
	alpha-BHC	90	360	0.012	NA
	beta-BHC	320	1300	0.037	NA
	BHC-Technical	320	1300	0.037	NA
	gamma-BHC (Lindane)	440	1700	0.052	NA
	Chlordane (technical)	1600	6500	0.19	NA
	4,4'-DDD	2400	10000	0.28	NA
	4,4'-DDE	1700	7000	0.2	NA
	4,4'-DDT	1700	7000	0.2	NA
	Dieldrin	30	11	0.0042	NA
	Endosulfan	370000	3700000	220	NA
	Endrin	18000	180000	11	NA
	Heptachlor	110	380	0.015	NA
	Heptachlor epoxide	53	190	0.0074	NA
	Methoxychlor	310000	3100000	180	NA
	Toxaphene	440	16000	0.061	NA
Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/PCDF)					
	2,3,7,8 - Tetrachlorodibenzo-p-dioxin (TCDD)	0.0039	0.016	0.00000045	NA

TABLE K.4.1

SOIL AND GROUNDWATER CLEANUP CRITERIA
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Compound*	*Soil PRGs Residential (mg/kg)*	*Soil PRGs Industrial (mg/kg)*	*PRG Tap Water (µg/L)*	*PRG Ambient Air (µg/M^3)*
TAL Metals (less earth metals)					
	Aluminum	76000	100000	36000	NA
	Antimony	31	410	15	NA
	Arsenic	0.39	1.6	0.045	NA
	Barium	5400	67000	2600	NA
	Beryllium	150	1900	73	NA
	Cadmium	37	450	18	NA
	Calcium	NA	NA	NA	NA
	Chromium Total	210	450	110	NA
	Cobalt	900	1900	730	NA
	Copper	3100	41000	1500	NA
	Iron	23000	100000	11000	NA
	Lead	400	800		NA
	Magnesium	NA	NA	NA	NA
	Manganese	1800	19000	880	NA
	Mercury	23	310	11	NA
	Nickel	1600	20000	730	NA
	Potassium	NA	NA	NA	NA
	Selenium	390	5100	180	NA
	Silver	390	5100	180	NA
	Sodium	NA	NA	NA	NA
	Thallium	5.2	67	2.4	NA
	Vanadium	78	1000	36	NA
	Zinc	23000	100000	11000	NA
	Cyanide, total	NA	NA	6.2	NA

		NESHAP (%)
Asbestos		
	ACM (by weight)	>1

Notes:

PRGs- USEPA Region 9 Preliminary Remediation Goals October 2004
NA - Not Available and/or Not Applicable (landfill gas sample VOC)
NESHAP- National Emission Standards for Hazardous Air Pollutants

TABLE K.4.2

ECOLOGICAL SCREENING OF GROUNDWATER
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Units*	*Ecological Screening Value*	*Reference (1)*
Volatiles			
1,1,1-Trichloroethane	µg/L	76	Ohio OMZA
1,1-Dichloroethane	µg/L	47	EPA Region V
1,2,4-Trimethylbenzene	µg/L	15	Ohio OMZA
1,2-Dichlorobenzene	µg/L	23	Ohio OMZA
1,2-Dichloroethane	µg/L	2000	Ohio OMZA
1,4-Dichlorobenzene	µg/L	9.4	Ohio OMZA
2-Butanone (MEK)	µg/L	22000	Ohio OMZA
4-Methyl-2-Pentanone (MIBK)	µg/L	170	EPA Region V
Acetone	µg/L	1700	EPA Region V
Benzene	µg/L	160	Ohio OMZA
Carbon disulfide	µg/L	15	Ohio OMZA
Chlorobenzene	µg/L	47	Ohio OMZA
Chloroethane	µg/L	1100	Mich. Chronic
Chloroform	µg/L	140	Ohio OMZA
Chloromethane	µg/L	--	--
cis-1,2-Dichloroethene	µg/L	970	Ohio OMZA
Cyclohexane	µg/L	--	--
Ethylbenzene	µg/L	61	Ohio OMZA
Methyl Tert Butyl Ether	µg/L	730	Ohio OMZA
Methylene chloride	µg/L	1900	Ohio OMZA
Tetrachloroethene	µg/L	53	Ohio OMZA
Toluene	µg/L	62	Ohio OMZA
trans-1,2-Dichloroethene	µg/L	970	Ohio OMZA
Trichloroethene	µg/L	220	Ohio OMZA
Vinyl chloride	µg/L	930	Ohio OMZA
Xylene (total)	µg/L	27	Ohio OMZA
Semi-Volatiles			
Acenaphthene	µg/L	9.4	Ohio OMZA
Acenaphthylene	µg/L	--	--
Acetophenone	µg/L	--	--
Anthracene	µg/L	0.35	EPA Region V
Atrazine	µg/L	--	--
Benzaldehyde	µg/L	--	--
Benzo(a)anthracene	µg/L	0.25	EPA Region V
Benzo(a)pyrene	µg/L	0.14	EPA Region V
Benzo(b)fluoranthene	µg/L	9.07	EPA Region V
Benzo(g,h,i)perylene	µg/L	7.64	EPA Region V
Benzo(k)fluoranthene	µg/L	--	--
Biphenyl	µg/L	--	--
4-Bromophenyl phenyl ether	µg/L	1.5	EPA Region V
Butyl benzylphthalate	µg/L	23	Ohio OMZA
Di-n-butylphthalate	µg/L	9.7	EPA Region V
Caprolactam	µg/L	--	--

TABLE K.4.2

ECOLOGICAL SCREENING OF GROUNDWATER
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Units*	*Ecological Screening Value*	*Reference (1)*
Carbazole	µg/L	--	--
4-Chloroaniline	µg/L	232	EPA Region V
bis(2-Chloroethoxy)methane	µg/L	19000	EPA Region V
bis(2-Chloroethyl)ether	µg/L	--	--
2,2'-oxybis(1-Chloropropane) (bis(2-chloroisopı	µg/L	--	--
4-Chloro-3-methylphenol	µg/L	--	--
2-Chloronaphthalene	µg/L	0.396	EPA Region V
2-Chlorophenol	µg/L	24	EPA Region V
4-Chlorophenyl phenyl ether	µg/L	--	--
Chrysene	µg/L	--	--
Dibenz(a,h)anthracene	µg/L	--	--
Dibenzofuran	µg/L	4	EPA Region V
3,3'-Dichlorobenzidine	µg/L	4.5	EPA Region V
2,4-Dichlorophenol	µg/L	11	EPA Region V
Diethyl phthalate	µg/L	110	EPA Region V
2,4-Dimethylphenol	µg/L	100	EPA Region V
Dimethyl phthalate	µg/L	--	--
4,6-Dinitro-2-methylphenol	µg/L	23	EPA Region V
2,4-Dinitrophenol	µg/L	19	EPA Region V
2,4-Dinitrotoluene	µg/L	44	Ohio OMZA
2,6-Dinitrotoluene	µg/L	81	Ohio OMZA
bis(2-Ethylhexyl)phthalate	µg/L	8.4	Ohio OMZA
Fluoranthene	µg/L	0.48	Ohio OMZA
Fluorene	µg/L	19	Ohio OMZA
Hexachlorobenzene	µg/L	0.0003	EPA Region V
Hexachlorobutadiene	µg/L	0.053	EPA Region V
Hexachlorocyclopentadiene	µg/L	77	EPA Region V
Hexachloroethane	µg/L	8	EPA Region V
Indeno(1,2,3-cd)pyrene	µg/L	4.31	EPA Region V
Isophorone	µg/L	920	Ohio OMZA
2-Methylnaphthalene	µg/L	330	EPA Region V
2-Methylphenol	µg/L	67	EPA Region V
4-Methylphenol	µg/L	53	Ohio OMZA
Naphthalene	µg/L	21	Ohio OMZA
2-Nitroaniline	µg/L	--	--
3-Nitroaniline	µg/L	--	--
4-Nitroaniline	µg/L	--	--
Nitrobenzene	µg/L	330	Ohio OMZA
2-Nitrophenol	µg/L	73	Ohio OMZA
4-Nitrophenol	µg/L	--	--
N-Nitrosodi-n-propylamine	µg/L	--	--
N-Nitrosodiphenylamine	µg/L	--	--
Di-n-octyl phthalate	µg/L	30	EPA Region V
Pentachlorophenol	µg/L	4.0	EPA Region V
Phenanthrene	µg/L	3.6	EPA Region V

TABLE K.4.2

ECOLOGICAL SCREENING OF GROUNDWATER
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Units*	*Ecological Screening Value*	*Reference (1)*
Phenol	µg/L	180	EPA Region V
Pyrene	µg/L	4.6	Ohio OMZA
2,4,5-Trichlorophenol	µg/L	--	--
2,4,6-Trichlorophenol	µg/L	4.9	Ohio OMZA
bis(2-Ethylhexyl)phthalate	µg/L	8.4	Ohio OMZA
Metals			
Aluminum	mg/L	0.087	USEPA NRWQC
Antimony	mg/L	0.19	Ohio OMZA
Arsenic	mg/L	0.15	Ohio OMZA
Barium	mg/L	0.22	Ohio OMZA
Beryllium	mg/L	0.0036	EPA Region V
Cadmium	mg/L	0.0073	Ohio OMZA
Calcium	mg/L	Nutrient	--
Chromium Total	mg/L	0.27	Ohio OMZA
Cyanide (total)	mg/L	0.0052	USEPA NRWQC
Iron	mg/L	1.00	USEPA NRWQC
Lead	mg/L	0.037	Ohio OMZA
Magnesium	mg/L	Nutrient	--
Manganese	mg/L	6.52	Mich. Chronic
Mercury	mg/L	0.00091	Ohio OMZA
Nickel	mg/L	0.17	Ohio OMZA
Potassium	mg/L	Nutrient	--
Sodium	mg/L	Nutrient	--
Thallium	mg/L	0.017	Ohio OMZA
Zinc	mg/L	0.39	USEPA NRWQC
Pesticides			
4,4'-DDE	µg/L	0.001	USEPA NRWQC
4,4'-DDT	µg/L	0.001	USEPA NRWQC
alpha-Chlordane	µg/L	0.0043	USEPA NRWQC
beta-BHC	µg/L	0.495	EPA Region V
delta-BHC	µg/L	667	EPA Region V
gamma-BHC (Lindane)	µg/L	0.057	Ohio OMZA
Heptachlor	µg/L	0.0038	USEPA NRWQC

Notes:

(1) Ohio OMZA: Ohio River Basin Aquatic Life and Human Health Tier I Criteria and Tier II Values, Outside Mixing Zone Area OAC 3745-1-32, July 27, 2005.
USEPA NRWQC: National Recommended Water Quality Criteria, EPA-822-R-02-047, Continuous Chronic Concentration, Office of Water, November 2002.
EPA Region V: Ecological Data Quality Levels, August 22, 2003. Available on the Internet at http://www.epa.gov/Region5/rcraca/edql.html

TABLE K.4.3

PERCENT RECOVERY AND RELATIVE PERCENT DIFFERENCE CONTROL LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*% Recovery Control Limits* [1]			
	Soil Sample		*Water Sample*	
Compound	*MS/MSD*	*LCS/LCD*	*MS/MSD*	*LCS/LCD*
Volatile Organic Compounds (VOC) [2]				
Acetone	10-200 (66)	58-130 (30)	45-128 (30)	22-200 (95)
Benzene	55-138 (20)	75-129 (20)	78-118 (20)	80-116 (20)
Bromodichloromethane	47-131 (51)	72-125 (30)	80-146 (30)	87-130 (30)
Bromoform	26-141 (64)	43-149 (30)	58-176 (30)	76-150 (30)
Bromomethane	15-152 (72)	24-152 (30)	55-145 (30)	64-129 (30)
2-Butanone	21-195 (60)	27-200 (46)	71-123 (30)	28-237 (65)
Carbon disulfide	27-149 (73)	50-137 (30)	69-138 (41)	73-139 (30)
Carbon tetrachloride	32-143 (68)	57-137 (30)	63-176 (30)	75-149 (30)
Chlorobenzene	49-139 (22)	75-127 (22)	76-117 (20)	76-117 (20)
Dibromochloromethane	44-135 (61)	49-135(30)	71-158 (30)	81-138 (30)
Chloroethane	32-140 (66)	31-144 (30)	59-142 (30)	66-126 (30)
Chloroform	59-128 (46)	73-115(30)	83-141 (30)	84-128 (30)
Chloromethane	28-130 (81)	15-136 (30)	40-137 (39)	48-123 (30)
Cyclohexane	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
1,2-Dibromo-3-chloropropane	50-150 (20)	50-150 (30)	70-130 (30)	70-130 (30)
1,2-Dibromoethane	50-150 (20)	50-150 (30)	70-130 (30)	70-130 (30)
1,2-Dichlorobenzene	50-150 (20)	50-150 (30)	70-130 (30)	70-130 (30)
1,3-Dichlorobenzene	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
1,4-Dichlorobenzene	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
Dichlorodifluoromethane	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
1,1-Dichloroethane	56-130 (54)	77-119 (30)	88-127 (30)	86-123 (30)
1,2-Dichloroethane	56-126 (38)	78-121 (30)	71-160 (30)	79-136 (30)
cis-1,2-Dichloroethene	48-127 (52)	77-114 (30)	87-114 (30)	85-113 (30)
trans-1,2-Dichloroethene	47-127 (58)	68-117 (30)	85-116(30)	80-120 (30)
1,1-Dichloroethene	43-147 (27)	55-142 (27)	62-130 (20)	63-130 (20)
1,2-Dichloropropane	54-125 (43)	78-116 (30)	87-114 (30)	82-115 (30)
cis-1,3-Dichloropropene	30-138 (49)	71-125 (30)	82-130 (30)	84-130 (30)
trans-1,3-Dichloropropene	34-134 (57)	67-125 (30)	73-147 (30)	84-130(30)
Ethylbenzene	36-133(72)	79-114 (30)	86-132 (30)	86-116 (20)
2-Hexanone	20-190(70)	29-200 (41)	81-128 (30)	35-200 (52)
Isopropylbenzene	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
Methyl acetate	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
Methylcyclohexane	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
Methylene chloride	45-129(49)	58-130 (30)	82-115 (30)	78-118 (30)
4-Methyl-2-pentanone	42-143 (60)	68-142 (60)	82-135 (30)	78-141 (32)
Methyl tert-butyl ether	70-130 (30)	70-130 (30)	70-130 (30)	70-130 (30)
Styrene	23-136 (65)	80-114 (30)	83-120 (30)	85-117 (30)
1,1,2,2-Tetrachloroethane	33-162 (90)	70-133 (30)	88-116 (30)	85-118 (30)
Tetrachloroethene	31-137 (81)	72-120 (30)	85-121 (30)	88-113 (30)
Toluene	46-147 (24)	71-130 (24)	70-119 (20)	74-119 (20)
1,2,4-Trichlorobenzene	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
1,1,1-Trichloroethane	48-132 (57)	67-123 (30)	71-162 (30)	78-140 (30)
1,1,2-Trichloroethane	58-128 (52)	82-116 (30)	86-129 (30)	83-122 (30)
Trichloroethene	46-143 (23)	70-131 (23)	62-130 (20)	75-122 (20)

TABLE K.4.3

PERCENT RECOVERY AND RELATIVE PERCENT DIFFERENCE CONTROL LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*% Recovery Control Limits* [1]			
	Soil Sample		*Water Sample*	
Compound	*MS/MSD*	*LCS/LCD*	*MS/MSD*	*LCS/LCD*
VOC Continued				
Trichlorofluoromethane	50-150 (20)	50-150 (20)	70-130 (30)	70-130 (30)
1,1,2-Trichloro-1,2,2-trifluoroethane	70-130 (30)	70-130 (30)	70-130 (30)	70-130 (30)
Vinyl chloride	30-136 (80)	24-152 (30)	88-126 (30)	61-120 (30)
Xylenes (total)	33-135 (78)	80-114 (30)	89-121 (30)	87-116 (30)
Semi-Volatile Organic Compounds (SVOC) [2]				
Acenaphthene	10-200 (30)	46-110 (30)	36-110 (30)	40-110 (30)
Acenaphthylene	10-200 (30)	47-110 (30)	39-110 (30)	43-110 (30)
Acetophenone	50-130 (30)	50-130 (30)	50-130 (30)	50-130 (30)
Anthracene	10-200 (30)	56-111 (30)	46-110 (30)	50-130 (30)
Atrazine	50-130 (30)	50-130 (30)	50-130 (30)	50-130 (30)
Benzaldehyde	10-130 (30)	10-130 (30)	10-130 (30)	10-130 (30)
Benzo(a)anthracene	10-200 (30)	58-111 (30)	52-110 (30)	55-115 (30)
Benzo(b)fluoranthene	10-200 (30)	43-124 (30)	33-114 (30)	43-122 (30)
Benzo(k)fluoranthene	10-200 (30)	38-122 (30)	32-121 (30)	43-124 (30)
Benzo(ghi)perylene	10-200 (30)	44-120 (30)	34-116 (30)	45-120 (30)
Benzo(a)pyrene	10-200 (30)	44-115 (30)	33-110 (30)	43-116 (30)
1,1'-Biphenyl	50-130 (30)	50-130 (30)	50-130 (30)	50-130 (30)
bis(2-Chloroethoxy)methane	36-110 (30)	42-110 (30)	35-110 (30)	39-110 (30)
bis(2-Chloroethyl) ether	32-118 (30)	41-110 (30)	27-110 (30)	34-113 (30)
bis(2-Ethylhexyl) phthalate	10-200 (30)	56-123 (30)	40-140 (30)	36-163 (30)
4-Bromophenyl phenyl ether	44-120 (30)	53-112 (30)	42-113 (30)	51-114 (30)
Butyl benzyl phthalate	43-138 (30)	57-121 (30)	51-121 (30)	53-126 (30)
Caprolactam	50-130 (30)	50-130 (30)	50-130 (30)	50-130 (30)
Carbazole	10-162 (30)	56-115 (30)	49-114 (30)	53-120 (30)
4-Chloroaniline	11-110 (30)	25-110 (30)	10-110 (30)	10-110 (30)
4-Chloro-3-methylphenol	32-117 (30)	42-110 (30)	33-110 (30)	39-110 (30)
2-Chloronaphthalene	40-110 (30)	46-110 (30)	34-110 (30)	39-110 (30)
2-Chlorophenol	32-110 (30)	39-110 (30)	26-110 (30)	27-110 (30)
4-Chlorophenyl phenyl ether	47-116 (30)	53-110 (30)	43-113 (30)	50-115 (30)
Chrysene	10-200 (30)	56-111 (30)	52-111 (30)	55-115 (30)
Dibenz(a,h)anthracene	10-200 (30)	45-122 (30)	35-118 (30)	46-122 (30)
Dibenzofuran	10-200 (30)	50-110 (30)	41-110 (30)	46-111 (30)
Di-n-butyl phthalate	31-145 (30)	57-119 (30)	50-117 (30)	55-122 (30)
3,3'-Dichlorobenzidine	10-110 (30)	31-110 (30)	10-110 (30)	19-110 (30)
2,4-Dichlorophenol	33-110 (30)	40-110 (30)	30-110 (30)	33-110 (30)
Diethyl phthalate	48-118 (30)	55-114 (30)	33-130 (30)	33-134 (30)
2,4-Dimethylphenol	19-114 (30)	28-110 (30)	11-110 (30)	12-110 (30)
Dimethyl phthalate	47-116 (30)	54-112 (30)	36-124 (30)	15-143 (30)
4,6-Dinitro-2-methylphenol	10-110 (30)	21-110 (30)	25-110 (30)	28-112 (30)
2,4-Dinitrophenol	10-110 (30)	10-110 (30)	11-119 (30)	17-112 (30)
2,4-Dinitrotoluene	42-118 (30)	55-116 (30)	46-119 (30)	52-123 (30)

TABLE K.4.3

PERCENT RECOVERY AND RELATIVE PERCENT DIFFERENCE CONTROL LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*% Recovery Control Limits* [1]			
	Soil Sample		*Water Sample*	
Compound	*MS/MSD*	*LCS/LCD*	*MS/MSD*	*LCS/LCD*
SVOC Continued				
2,6-Dinitrotoluene	28-137 (30)	54-115 (30)	48-115 (30)	52-119 (30)
Di-n-octyl phthalate	10-182 (30)	45-123 (30)	36-134 (30)	44-128 (30)
Fluoranthene	10-200 (30)	55-118 (30)	53-111 (30)	54-122 (30)
Fluorene	10-187 (30)	51-110 (30)	43-110 (30)	47-112 (30)
Hexachlorobenzene	37-122 (30)	51-110 (30)	40-113 (30)	51-112 (30)
Hexachlorobutadiene	30-110(30)	39-110 (30)	14-110 (30)	13-110 (30)
Hexachlorocyclopentadiene	10-110 (30)	10-110 (30)	10-110 (30)	10-110 (30)
Hexachloroethane	13-110 (30)	38-110 (30)	10-110 (30)	12-110 (30)
Indeno(1,2,3-cd)pyrene	10-200 (30)	45-121 (30)	36-116 (30)	46-121 (30)
Isophorone	32-129 (30)	46-117 (30)	34-125 (30)	44-128 (30)
2-Methylnaphthalene	10-200 (30)	46-110 (30)	35-110 (30)	35-110 (30)
2-Methylphenol	19-124 (30)	36-110 (30)	26-110 (30)	30-110 (30)
4-Methylphenol	27-116 (30)	40-110 (30)	25-110 (30)	32-110 (30)
Naphthalene	10-200 (30)	42-110 (30)	32-110 (30)	31-110 (30)
2-Nitroaniline	31-141 (30)	47-124 (30)	31-129 (30)	43-130 (30)
3-Nitroaniline	24-110 (30)	44-110 (30)	23-112 (30)	45-116 (30)
4-Nitroaniline	23-124 (30)	50-110 (30)	26-115 (30)	45-120 (30)
Nitrobenzene	33-111 (30)	40-110 (30)	26-118 (30)	37-115 (30)
2-Nitrophenol	37-110 (30)	35-110 (30)	30-110 (30)	29-110 (30)
4-Nitrophenol	10-125 (30)	24-117 (30)	13-127 (30)	12-130 (30)
N-Nitrosodiphenylamine	10-169 (30)	54-112 (30)	28-118 (30)	53-113 (30)
N-Nitrosodi-n-propylamine	30-121 (30)	40-114 (30)	25-119 (30)	37-121 (30)
2,2'-oxybis(1-Chloropropane)	25-124 (30)	36-116 (30)	13-124 (30)	25-128 (30)
Pentachlorophenol	10-182 (30)	10-110 (30)	23-110 (30)	26-110 (30)
Phenanthrene	10-200 (30)	54-110 (30)	47-110 (30)	52-114 (30)
Phenol	10-144 (30)	39-110 (30)	16-110 (30)	14-112 (30)
Pyrene	10-200 (30)	58-113 (30)	54-115 (30)	55-120 (30)
2,4,5-Trichlorophenol	32-112 (30)	42-110 (30)	36-110 (30)	39-110 (30)
2,4,6-Trichlorophenol	22-110 (30)	37-110 (30)	34-110 (30)	35-110 (30)
Polychlorinated Biphenyls [2,3]				
Aroclor 1016	10-199 (30)	34-127 (30)	10-166 (30)	44-119 (30)
Aroclor 1260	10-199 (30)	32-141 (30)	21-140 (30)	41-118 (30)
Pesticides				
Aldrin	10-199(30)	25-149(30)	21-159 (30)	54-131 (30)
alpha-BHC	10-182(30)	19-155(30)	17-180 (30)	50-145 (30)
beta-BHC	10-199(30)	18-160 (30)	21-182 (30)	50-147 (30)
delta-BHC	10-199(30)	17-169(30)	32-183 (30)	51-157 (30)
gamma-BHC (Lindane)	10-199(30)	21-155(30)	23-177 (30)	51-145 (30)
alpha-Chlordane	10-199(30)	22-151(30)	24-165 (30)	51-137 (30)
gamma-Chlordane	10-199(30)	21-160(30)	22-174 (30)	53-142 (30)

TABLE K.4.3

PERCENT RECOVERY AND RELATIVE PERCENT DIFFERENCE CONTROL LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*% Recovery Control Limits* [1]			
	Soil Sample		*Water Sample*	
Compound	*MS/MSD*	*LCS/LCD*	*MS/MSD*	*LCS/LCD*
Pesticides Continued				
4,4'-DDD	10-199(30)	15-176(30)	10-199 (30)	44-163 (30)
4,4'-DDE	10-199(30)	23-152(30)	10-180 (30)	50-137 (30)
4,4'-DDT	10-199(30)	13-152(30)	10-185 (30)	43-148 (30)
Dieldrin	10-199(30)	24-152(30)	33-167 (30)	54-139 (30)
Endosulfan I	10-199(30)	10-112(30)	10-122 (30)	10-112 (30)
Endosulfan II	10-127(30)	10-113(30)	10-120 (30)	10-114 (30)
Endosulfan sulfate	10-167(30)	16-154(30)	42-165 (30)	50-142 (30)
Endrin	10-199(30)	21-156(30)	18-189 (30)	49-145 (30)
Endrin aldehyde	10-199(30)	17-156(30)	31-169 (30)	50-140 (30)
Endrin ketone	10-199(30)	22-147(30)	10-199 (30)	48-141 (30)
Heptachlor	10-199(30)	24-147(30)	10-199 (30)	47-141 (30)
Heptachlor epoxide	10-199(30)	21-151(30)	40-157 (30)	49-140 (30)
Methoxychlor	10-199(30)	14-160(30)	26-177 (30)	46-147 (30)
Toxaphene	NA	NA	NA	NA
Herbicides				
2,4-Dichlorophenoxyacetic acid (2,4-D)	15-110 (30)	33-110 (30)	--	--
2,4,5-Trichlorophenoxyacetic acid (2,4,5-TP)	10-117 (30)	42-110 (30)	--	--
PCDD/PCDF				
2,3,7,8 - TCDD	77-133(20)	77-133(20)	71-128 (20)	71-128 (20)
2,3,7, 8 - TCDF	80-146(20)	80-146(20)	75-142 (20)	75-142 (20)
1,2,3,7,8 - PeCDD	74-145(20)	74-145(20)	74-139 (20)	74-139 (20)
1,2,3,7,8 - PeCDF	84-143(20)	84-143(20)	80-140 (20)	80-140 (20)
2,3,4,7,8 - PeCDF	76-157(20)	76-157(20)	71-144 (20)	71-144 (20)
1,2,3,4,7,8 - HxCDD	68-146(20)	68-146(20)	65-144 (20)	65-144 (20)
1,2,3,6,7,8 - HxCDD	79-141(20)	79-141(20)	73-142 (20)	73-142 (20)
1,2,3,7,8,9 - HxCDD	68-139(20)	68-139(20)	60-147 (20)	60-147 (20)
1,2,3,4,7,8 - HxCDF	78-141(20)	78-141(20)	64-149 (20)	64-149 (20)
1,2,3,6,7,8 - HxCDF	78-144(20)	78-144(20)	56-161 (20)	56-161 (20)
1,2,3,7,8,9 - HxCDF	70-144(20)	70-144(25)	53-163 (20)	53-163 (20)
2,3,4,6,7,8 - HxCDF	73-157(20)	73-157(20)	60-169 (20)	60-169 (20)
1,2,3,4,6,7,8 - HpCDD	74-147(20)	74-147(20)	79-137 (20)	79-137 (20)
1,2,3,4,6,7,8 - HpCDF	79-143(20)	79-143(20)	78-141 (20)	78-141 (20)
1,2,3,4,7,8,9 - HpCDF	79-150(20)	79-150(20)	80-146 (20)	80-146 (20)
OCDD	75-153(20)	75-153(20)	71-147 (20)	71-147 (20)
OCDF	70-158(20)	70-158(20)	76-147 (20)	76-147 (20)

TABLE K.4.3

PERCENT RECOVERY AND RELATIVE PERCENT DIFFERENCE CONTROL LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*% Recovery Control Limits* [1]			
	Soil Sample		*Water Sample*	
Compound	*MS/MSD*	*LCS/LCD*	*MS/MSD*	*LCS/LCD*
TAL Inorganics				
Aluminum	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Antimony	75-125 (20)	80-120 (20)	44-153 (20)	57-110(20)
Arsenic	23-131(20)	73-110(20)	82-123 (20)	86-118 (20)
Barium	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Beryllium	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Cadmium	75-125 (20)	80-120 (20)	78-117 (20)	89-114 (20)
Calcium	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Chromium	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Cobalt	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Copper	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Iron	75-125 (20)	77-122 (20)	75-125 (20)	80-120 (20)
Lead	75-125 (20)	80-120 (20)	73-115 (20)	84-113 (20)
Magnesium	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Manganese	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Mercury	11-192 (20)	73-121 (20)	69-134 (20)	81-123(20)
Nickel	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Potassium	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Selenium	75-125 (20)	80-120 (20)	72-148 (20)	90-128 (20)
Silver	75-125 (20)	80-120 (20)	10-139 (20)	83-111 (20)
Sodium	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Thallium	62-110 (20)	71-110 (20)	69-117 (20)	82-113 (20)
Vanadium	75-125 (20)	80-120 (20)	75-125 (20)	80-120 (20)
Zinc	75-125 (20)	80-120 (20)	49-156 (20)	90-129 (20)
Cyanide, Total	50-134 (20)	68-123 (20)	42-140 (20)	69-118 (20)
MNA Inorganics				
Alkalinity	NA	NA	10-160 (24)	90-127 (20)
Chloride	NA	NA	80-120 (20)	90-110 (20)
Dissolved Organic Carbon (1)	NA	NA	72-136 (20)	88-115 (20)
Hardness, total	NA	NA	NA	NA
Hardness, carbonate	NA	NA	NA	NA
Nitrate	NA	NA	80-120 (20)	90-110 (20)
Nitrite	NA	NA	80-120 (20)	90-110 (20)
Sulfate	NA	NA	80-120 (20)	90-110 (20)
Sulfide	NA	NA	75-125 (20)	75-125 (20)
MNA Dissolved Gases				
Methane	NA	NA	74-138 (30)	74-138 (30)
Ethane	NA	NA	73-140 (30)	73-140 (30)
Ethene	NA	NA	75-127 (30)	75-127 (30)
Asbestos	NA	[3]	NA	NA

Notes:

[1] Values in parenthesis are the maximum relative percent difference (RPD) values allowed for MS/MSD, or LCS/LCD analyses. Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed, identified in the laboratory's report will be used for data validation purposes.

[2] The laboratory may prepare and analyze an "all-analyte" spike; however, the control analytes presented are utilized for QC batch control.

[3] Within one quantitation range
NA - not applicable

TABLE K.4.4

SURROGATE COMPOUND PERCENT RECOVERY CONTROL LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

		% Recovery Control Limits[1]		
Parameter	*Surrogate Compound*	*Soil*	*Water*	*Air*
Volatile Organics Compounds				
	4-Bromofluorobenzene	47-158	74-116	70-130
	Dibromofluoromethane	59-138	73-122	NA
	1,2-Dichloroethane-d4	61-130	61-128	70-130
	Toluene-d8	60-143	76-110	70-130
Semi-volatile Organic Compounds				
	Nitrobenzene-d_5	24-112	27-111	--
	2-Fluorobiphenyl	34-110	28-110	--
	Terphenyl-d_{14}	41-119	37-119	--
	2-Fluorophenol	26-110	10-110	--
	2,4,6-Tribromophenol	10-118	22-120	--
	Phenol-d_5	28-110	10-110	--
Polychlorinated Biphenyls				
	Decachlorobiphenyl	10-127	10-199	--
	Tetrachloro-meta-xylene	27-130	10-196	--
Pesticides				
	Decachlorobiphenyl	10-199	10-139	--
	Tetrachloro-meta-xylene	10-199	25-142	--
Herbicides				
	2,4-Dichlorophenylacetic acid	19-122	--	--
	Internal Standards			
PCDD/PCDF[2]	^{13}C-2,3,7,8 - TCDD	40-135	40-135	--
	^{13}C-1,2,3,7,8 - TCDF	40-135	40-135	--
	^{13}C-1,2,3,7,8 - PeCDD	40-135	40-135	--
	^{13}C-1,2,3,7,8 - PeCDF	40-135	40-135	--
	^{13}C-1,2,3,6,7,8 - HxCDD	40-135	40-135	--
	^{13}C-1,2,3,4,7,8 - HxCDF	40-135	40-135	--
	^{13}C-1,2,3,4,6,7,8 - HpCDD	40-135	40-135	--
	^{13}C-1,2,3,4,6,7,8 - HpCDF	40-135	40-135	--
	^{13}C-OCDD	40-135	40-135	--

Notes:

[1] Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed will be used for data validation purposes.

[2] Surrogates identified are actually isotopically labeled internal standards.

TABLE K.5.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Instrument/Equipment	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Gas Chromatograph/Mass Spectrometer (GC/MS)	1. Replace pump oil as needed. 2. Change septa weekly or as often as needed. 3. Change gas line dryers as needed. 4. Replace electron multiplier as often as needed. 5. Replace gas jet splitter as needed. 6. Replace GC injector glass liner weekly or as often as needed. 7. Replace GC column as needed. 8. Check daily to ensure that gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 9. Check daily to ensure the pressure on the primary regulator never runs below 100 psi. 10. Clean source as needed.	1. Syringes 2. Septa 3. Various electronic components 4. Glass jet splitter 5. GC column 6. Glass liners
Gas Chromatograph	1. Change septa weekly or as often as needed. 2. Change gas line dryers as needed. 3. Replace GC injector glass liner weekly or as often as needed. 4. Replace GC column as needed. 5. Clean/replace GC detector as needed. 6. Check daily to ensure that gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 7. Check daily to ensure the pressure on the primary regulator never run below 100 psi.	1. Syringes 2. Septa 3. Detectors 4. Glass liner 5. GC column

TABLE K.5.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Instrument/Equipment	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Purge and Trap Sample Concentrator	1. Replace trap as needed. 2. Decontaminate the system after running high concentration samples or as required by blank analysis. 3. Leak check system daily and as often as needed 4. Check daily to ensure the gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 5. Check daily to ensure the pressure on the primary regulator never run below 100 psi.	1. Spare traps 2. Spare sparger vessels 3. Various electronic components/ circuits 4. Plumbing supplies - tubing, fittings
Mercury Analyzer	1. Clean tubing and quartz cell weekly or as often as needed. 2. Clean aspirator after each batch samples or as necessary. 3. Check daily to ensure the gas supply is sufficient for the day's activity, and the deliver pressures are set as described in the SOP.	1. Quartz cells 2 Aspirator
Inductively Coupled Plasma Spectrometer (ICP)	1. Clean torch assembly and mixing chamber when discolored or after eight hours of running high dissolved solid samples 2. Clean nebulizer as needed. 3. Check daily to ensure the gas supply is sufficient for the day's activity pressures are set as described in the SOP.	1. Spare torch and mixing chambers 2. Spare nebulizer 3. Spare capillary tubing

TABLE K.5.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Instrument/Equipment	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
ICP/Mass Spectrometer (MS)	1. Change the peristaltic pump tubing 2. Inspect the sampler and skimmer cones for cleanliness, clean if necessary. 3. Check the vacuum system oil levels 4. Rinse nebulizer with 1% HNO_3 for five minutes 5. Clean torch assembly and mixing chamber when discolored	1. Peristaltic pump tubing 2. Spare torch and mixing chambers 3. Spare nebulizer 4. Spare capillary tubing
Autoanalyzer/Spectrophotometer	1. Inspect pump tubes after each 8-hour run; replace if discolored or distorted 2. Check daily to ensure the gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP.	1. Pump tubing 2. Colorimeter lamps
pH Meter	1. Check battery (if used in field); and replace if discharged. 2. After use in samples containing free oil, wash the electrode in soap and rinse thoroughly with water. Immerse the lower third of the electrode in diluted HCl (1:9) solution for 10 minutes to remove any film formed. Rinse thoroughly with water. 3. Keep electrode properly filled with appropriate filling electrolyte solution.	1. Standard buffers 2. Electrolyte filling solution 3. Spare electrode
Refrigerators	1. Monitor temperature twice daily.	
Ovens	1. Monitor temperature daily.	

TABLE K.5.2

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2]	*Volume of Sample*	*Shipping*	*Normal Packaging*
WATER (Groundwater/Surface Water)						
VOC	Three 40 mL teflon-lined septum vials per analysis	HCl to pH < 2 Iced, 4 ± 2° C	14 days for analysis	Fill completely, no air bubbles	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
PCB, SVOC, Pesticides	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	7 days for extraction 40 days after extraction for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Metals	One 1 liter plastic bottle	HNO_3 to pH < 2 Iced, 4 ± 2° C	180 days (mercury-28 days) for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Cyanide (total)	One 250 ml plastic bottle	NaOH to pH>12 Iced, 4 ± 2° C	14 days for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Hardness (calculated)	One 250 ml plastic bottle	HNO_3 to pH < 2 Iced, 4 ± 2° C	180 days (mercury-28 days) for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Alkalinity	One 500-ml plastic bottle	Iced, 4 ± 2° C	14 days for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Nitrate	One 250-ml plastic bottle	Iced, 4 ± 2° C	48 hours to start analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Nitrite	One 250-ml plastic bottle	Iced, 4 ± 2° C	48 hours to start analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips

TABLE K.5.2

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2]	*Volume of Sample*	*Shipping*	*Normal Packaging*
WATER (Groundwater, Surface Water and Wastewater) (Cont'd)						
Sulfate	One 250-ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Sulfide	One 250-ml plastic bottle	Zinc Acetate/ NaOH to pH>9 Iced, 4 ± 2° C	7 days for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips
DOC	Two 40 ml Teflon-lined septum vials per analysis	H2SO4 to pH < 2 Iced, 4 ± 2° C	28 days for analysis	Fill completely, no air bubbles	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Dissolved Gases	Three 40 mL teflon-lined septum vials per analysis	HCl to pH < 2 Iced, 4 ± 2° C	14 days for analysis	Fill completely, no air bubbles	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
TCLP VOC	One 250 ml glass bottle	Iced, 4 ± 2° C	14 days for TCLP and 14 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
TCLP SVOC, TCLP Pesticides, TCLP Herbicides	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	7 days for TCLP, 7 days for preparation and 40 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
TCLP Metals	Two 1 liter amber glass bottles	Iced, 4 ± 2° C	180 days (mercury 28 days) for TCLP and analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Corrosivity, Ignitibility	One 1 liter plastic bottle	Iced, 4 ± 2° C	14 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips

TABLE K.5.2

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2]	*Volume of Sample*	*Shipping*	*Normal Packaging*
SOLID (Soil/Sediment)						
VOC[3,4]	Three 5g En Core Sampler™ per sample	Iced, 4 ± 2° C	48 hours for extraction 14 days for analysis	Fill completely	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Pesticides, PCB, SVOC, Herbicides	Two 4-ounce glass jars	Iced, 4 ± 2° C	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Metals	One 4-ounce glass jar	Iced, 4 ± 2° C	180 days (mercury 28 days) for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Cyanide (total)	One 4-ounce glass jar	Iced, 4 ± 2° C	14 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
TCLP VOC	One 4-ounce glass jar	Iced, 4 ± 2° C	14 days for TCLP and 14 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
TCLP SVOC	One 4-ounce glass jar	Iced, 4 ± 2° C	14 days for TCLP, 7 days for preparation and 40 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
TCLP Metals	One 4-ounce glass jar	Iced, 4 ± 2° C	180 days (mercury 28 days) for TCLP and analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Corrosivity	One 4-ounce glass jar	Iced, 4 ± 2° C	14 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips

TABLE K.5.2

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Analyses	*Sample Containers* [1]	*Preservation*	*Maximum Holding Time from Sample Collection* [2]	*Volume of Sample*	*Shipping*	*Normal Packaging*
SOLID (Soil/Sediment) (Cont'd)						
Ignitibility	One 4-ounce glass jar	Iced, 4 ± 2° C	14 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
PCDD/PCDF	One 4-ounce glass jar	Iced, 4 ± 2° C	30 days for extraction 45 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
Asbestos	One taped &sealed ziploc bag	None	None	Fill bag	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
AIR (Soil Gas)						
VOC	One 6-L Summa Canister	None	14 days for analysis	Fill canister maintaining slight negative pressure	Overnight or Hand Deliver	Cardboard Shipper

Notes:

[1] - Multiple parameters on a single sample with identical preservation requirements may be combined into one single sample container.

[2] - These are technical holding times, i.e., are based on time elapsed from time of sample collection.

[3] - If Encore™ samples cannot be analyzed within 48 hours, they can be frozen at -10 degrees Celsius.

[4] - If no other samples are submitted with solid VOCs a separate container must be included for percent moisture.

TABLE K.5.3

SUMMARY OF ANALYTICAL METHODS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter[1]	*Preparation Method*[2]	*Laboratory Preparation SOP*	*Analytical Method*[2]	*Laboratory Analytical SOP*
Water Samples (Groundwater/Surface Water)				
VOC	SW-846 5030B	CORP-MS-0002NC	SW-846 8260B	CORP-MS-0002NC
SVOC	SW-846 3500 series	CORP-OP-0001NC	SW-846 8270C	CORP-MS-0001NC
PCB	SW-846 3500 series	NC-OP-0032	SW-846 8082	NC-GC-038
Pesticides	SW-846 3500 series	NC-OP-0032	SW-846 8081	NC-GC-038
Metals[3]				
ICP Metals	SW-846 3010A/3020A	CORP-IP-0003	SW-846 6010B	NC-MT-0012
ICP/MS Metals	SW-846 3010A/3020A	CORP-IP-0003	SW-846 6020	NC-MT-0002
Mercury	SW-846 7470A	CORP-MT-0005NC	SW-846 7470A	CORP-MT-0005NC
Cyanide (total)	SW-846 9012A	NC-WC-0032	SW-846 9012A	NC-WC-0031
Alkalinity	SM 2320B	NC-WC-0006	SM 2320B	NC-WC-0003
Chloride	SW-846 9056	NC-WC-0084	SW-846 9056	NC-WC-0084
DOC	SW-846 9060	NC-WC-0017	SW-846 9060	NC-WC-0017
Hardness, total	SM 2340B	NC-WC-0036	SM 2340B	NC-WC-0036
Hardness, carbonate	SM 2340B	NC-WC-0036	SM 2340B	NC-WC-0036
Nitrate	SW-846 9056	NC-WC-0084	SW-846 9056	NC-WC-0084
Nitrite	SW-846 9056	NC-WC-0084	SW-846 9056	NC-WC-0084
Sulfate	SW-846 9056	NC-WC-0084	SW-846 9056	NC-WC-0084
Sulfide	SW-846 9030A	NC-WC-0060	SW-846 9030A	NC-WC-0060
Dissolved Gases	EPA SOP RSK 175	NC-GC-0032	EPA SOP RSK 175	NC-GC-0032
Solid Samples (Soil/Sediment)				
VOC	SW-846 5035	CORP-MS-0002NC	SW-846 8260B	CORP-MS-0002NC
SVOC	SW-846 3500 series	CORP-OP-0001NC	SW-846 8270C	CORP-MS-0001NC
PCB	SW-846 3500 series	NC-OP-0032	SW-846 8082	NC-GC-038
Pesticides	SW-846 3500 series	NC-OP-0032	SW-846 8081	NC-GC-038
Herbicides	SW-846 3500 series	NC-OP-0031	SW-846 8150A	NC-GC-038
Metals[3]				
ICP Metals	SW-846 3050 B	CORP-IP-0002NC	SW-846 6010B	CP-MT-012
ICP/MS Metals	SW-846 3050B	CORP-IP-0002NC	SW-846 6020	NC-MT-0002
Mercury	SW-846 7471A	CORP-MT-011	SW-846 7471A	CORP-MT-011
Cyanide (total)	SW-846 9012A	NC-WC-0032	SW-846 9012A	NC-WC-0031
Sulfide (total)	SW-846 9012A	NC-WC-0032	SW-846 9012A	NC-WC-0031
PCDDs/PCDFs	SW-846 8290	WS-ID-0005	SW-846 8290	WS-ID-0005
Asbestos	CARB 435	EML 100217	CARB 435	EML 100217

TABLE K.5.3

SUMMARY OF ANALYTICAL METHODS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter[1]	*Preparation Method*[2]	*Laboratory Preparation SOP*	*Analytical Method*[2]	*Laboratory Analytical SOP*
Air Samples (Soil Gas)				
VOC	EPA TO-14A	COI-MS-0003	EPA TO-14A	COI-MS-0003
Waste Characterization				
TCLP	SW-846 1311	CORP-IP-0004NC	NA	NA
VOC	SW-846 5030B	CORP-MS-0002NC	SW-846 8260B	CORP-MS-0002NC
SVOC	SW-846 3520C	CORP-OP-0001NC	SW-846 8270C	CORP-MS-0001NC
Metals				
ICP Metals	SW-846 3010A	CORP-IP-0002NC	SW-846 6010B	CP-MT-012
mercury	SW-846 7470A	CORP-MT-011	SW-846 7470A	CORP-MT-011
PCB (solids)	SW-846 3550B	NC-OP-0032	SW-846 8082	NC-GC-038
PCB (waters)	SW-846 3500C	NC-OP-0032	SW-846 8082	NC-GC-038
Corrosivity	NA	NA	SW-846 9045	NC-WC-0010
Ignitibility (flashpiont)	NA	NA	SW-846 1010	NC-WC-0034
Cyanide	SW-846 9012A	NC-WC-0032	SW-846 9012A	NC-WC-0031
Sulfide	SW-846 9030A	NC-WC-0060	SW-846 9030A	NC-WC-0060

Notes:

1 Refer to Tables 3.2, 3.3, 3.4, 3.5 for the compounds/elements of each parameter group.

2 Preparation and Analytical Method References:
- SW-846 - "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods ", SW-846, 3rd Edition, and Promulgated Updates, November 1986.
- EPA-WW - "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, Revised March 1983.
- SM - "Standard Methods for the Examination of Water and Wastewater", APHA, AWWA & WEF, 19th Edition, 1995
- RSKSOP-175 - U.S.EPA Robert S. Kerr Environmental Research Laboratory, Ada OK, Standard Operating Procedure
- EPA 600 - "Sample Preparation and Analysis for Asbestos and Other Fibers by Poarized Light Microscopy (PLM)", EPA Method 600.R-93/116.
- CARB 435 - "California Air Resource Board Method 425", EPA-600/M4-82-020 December 1982/EPA-600/R-93/116

3 Metals by Method - Aluminum and Iron will be analyzed by the most appropriate method depending on sample concentrations.

Water ICP:	Aluminum, Barium, Beryllium, Chromium, Cobalt, Copper, Iron, Magnesium, Manganese, Nickel, Vanadium.
Water ICP/MS:	Antimony, Arsenic, Cadmium, Lead, Selenium, Silver, Thallium, Zinc.
Soil ICP:	Aluminum, Antimony, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Manganese, Nickel, Selenium, Silver, Vanadium, Zinc.
Soil ICP/MS:	Arsenic, Thallium.

VOC = Volatile Organic Compounds
SVOC = Semi-volatile Organic Compounds
PCB = Polychlorinated Biphenyls
ICP = Inductively Coupled Plasma
ICP/MS = Inductively Coupled Plasma/Mass Spectrometer
DOC = Dissolved Organic Carbon

TABLE K.5.4

DATA REVIEW AND VALIDATION LEVELS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Item Reviewed	*Reduced Data Validation*	*Full Data Validation*
General Report Deliverables		
Methods/Procedures	X	X
Parameter List	X	X
Report/Detection Limits	X	X
Documentation/Deliverables	X	X
Sample Specific and Batch QC Data		
Sample Preservation and Holding Times	X	X
Method Blanks	X	X
Field Blanks (Trip and Rinsate Blanks)	X	X
System Monitoring Compounds (Surrogates)	X	X
MS/MSD - Organics	X	X
MS/MSD, MS/MD - Inorganics	X	X
Laboratory Control Sample (LCS)	X	X
Field Duplicates	X	X
Expanded Data Elements		
Instrument Performance Check (GC/MS & ICP/MS)		X
Initial Calibration - Organics		X
Continuing Calibration - Organics		X
Initial Calibration Verification - Inorganics		X
Continuing Calibration Verification - Inorganics		X
Internal Standards (GC/MS & ICP/MS)		X
Instrument Blanks - Inorganics		X
ICP/MS Internal Standards		X
ICP Interference Check Samples		X
Serial Dilutions		X
Compound Identification [1]		X
Chromatography [1]		X
Compound/Analyte Quantitation (raw data) [1]		X
Report Limit Verification [1]		X

Note:

[1] Raw data review including calculation checks and chromatography review will be completed on 10 percent of the sample data unless data warrents otherwise.

APPENDIX K-A

SITE HISTORY

K-A 1.1 SITE HISTORY

Landfill operations continued in the central portion of the Site until the death of the landfill's operator, Mr. Alcine Grillot, in 1996. The current owners of the properties located within the Site are Valley Asphalt, Jim City Salvage, MCD, Ronald Barnett, Kathryn A. Boesch and Margaret C. Grillot. Most of the northern portion of the Site is owned by Valley Asphalt.

K-A 1.2 PREVIOUS INVESTIGATIONS

The purpose of this section is to present a discussion of previous investigations related to the Site. This background information is required to support subsequent sections of this QAPP.

The following investigations have been conducted at the Site since 1985:

- Ohio EPA, 1985, Preliminary Assessment for the South Dayton Dump and Landfill;
- Ecology and Environment, Inc. (EEI), 1991, Screening Site Inspection Report for South Dayton Dump, Moraine, Ohio. Prepared by EEI on behalf of USEPA;
- PRC Environmental Management, Inc. (PRC), 1995, Focused Site Inspection Prioritization Site Evaluation Report for the South Dayton Dump;
- PSARA Technologies, Inc. (PSARA), 1996, Installation of Groundwater Monitoring Wells at the South Dayton Dump, Moraine, Ohio. Prepared by PSARA on behalf of Ohio EPA;
- Ohio EPA, 1996, Site Team Evaluation Prioritization Report, South Dayton Dump and Landfill;
- PFI, 1998-2005. Groundwater monitoring well installations, groundwater sampling, analyses, and water level measurements;
- TCA Environmental, 2000, Environmental Remediation Report at Valley Asphalt. Prepared for Valley Asphalt; and
- Memo from Ohio EPA to USEPA dated January 24, 2006, regarding "South Dayton Dump, Valley Asphalt Reconnaissance Brief".

Figure K-3.3 shows the locations of the existing groundwater monitoring wells installed in and around the Site. Figure K-3.2 shows the location of the historical soil samples and boreholes collected as part of the investigations listed above.

K-A 1.3 1985 OHIO EPA PRELIMINARY ASSESSMENT (PA)

The 1985 Ohio EPA investigation that consisted of an aerial inspection of the Site and interviews made the following conclusions and recommendations:

- The presence/disposal of hazardous chemicals at the Site posed a potential threat to groundwater beneath the Site, and to the GMR;
- Groundwater flow is to the west toward the GMR[1]; and
- Ohio EPA rated the Site as a high priority for State and Federal action, and recommended the installation of groundwater monitoring wells.

K-A 1.4 1991 EEI SCREENING SITE INSPECTION (SSI)

The 1991 EEI SSI investigation was completed on behalf of USEPA. The SSI consisted of the collection and analysis of surface and subsurface soil samples from the Site.

EEI collected nine surface and two subsurface (1-foot depth) soil samples and analyzed the samples for VOCs, PAHs, PCBs, and metals. EEI concluded that each of these types of analytes was detected at concentrations above background. Analytical results, are presented in Table 2.2 of the draft RI/FS Work Plan, and are summarized as follows:

- *Chlorinated solvents (200 µg/Kg 1,2-DCE [1,2-dichloroethene], 4 µg/Kg TCE [trichloroethene] and 11 µg/Kg PCE [tetrachloroethene]) in surface soil sample S8 in eastern central area of Site on north side of ravine.*
- *Highest levels of PCBs (4.2 mg/Kg Aroclor 1248 and 2.8 mg/Kg Aroclor 1260 in surface soil sample S2 at edge of water-filled Large Pond and in vicinity of area where Alcine reportedly dismantled transformers from DP&L.*

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1 Based on CRA's review it appears that this determination was not made on the basis of monitoring well information — no wells were present at the time. Subsequent information collected by others during water level monitoring at new wells conflicts with this interpretation.

- *Highest SVOCs and PAHs in surface soil sample S3 south of north access road in center of Site near deteriorated drum (90 mg/Kg total SVOCs) and S6 along western edge of central Site area 450 to 500 feet east of river (95 mg/Kg total SVOCs).*
- *Highest levels of inorganic chemicals generally in S3 and S8 (lead as high as 3,300 mg/Kg, copper as high as 2,200 mg/Kg, cadmium as high as 14 mg/Kg, mercury as high as 0.31 mg/Kg, and nickel as high as 402 mg/Kg).*
- *Highest level of arsenic in S9 (69 mg/Kg) in central area of Site north of ravine.*
- *Elevated OVA [organic vapor analyzer] readings detected near opening of former air curtain destructor.*

The locations of the samples shown on Figure K-3.4 are based on scanned copies of the report. Survey data for these sample locations are not available.

K-A 1.5 1995 PRC FOCUSED SITE INSPECTION PRIORITIZATION (FSIP)

The 1995 FSIP consisted of a Site inspection, a review of available information and evaluation of the potential threat to human health and the environment posed by the Site, and the development of recommendations to assess the Site further. The FSIP recommended that groundwater monitoring wells be installed and sampled and surface water samples be collected and analyzed.

K-A 1.6 1996 PSARA MONITORING WELL INSTALLATION

The 1996 PSARA report was completed on behalf of Ohio EPA. PSARA installed seven soil borings and temporary monitoring wells along the south-central, southwestern, western, and northwestern portions of the Site. PSARA collected soil samples for lithologic description and field screening. Methane was detected in the sample headspace at five boring locations. PSARA reported that a flame ionization detector (FID) reading of over 1,000 parts per million (ppm) was measured at one location. Field data are summarized in Table 2.4 of the draft RI/FS Work Plan.

The investigation included the collection of groundwater samples from the soil borings. The samples were analyzed for VOCs. The concentrations of all VOCs detected were below federal maximum contaminant levels (MCLs) for drinking water. The

groundwater analytical results are summarized in Table 2.5 of the draft RI/FS Work Plan.

PSARA also installed three permanent groundwater monitoring wells in locations that were based on access constraints and the presumed historical groundwater flow direction. A monitoring well at the Dayton Power and Light facility to the east of the Site was also utilized and considered a background location, but its location was not surveyed. The stratigraphic and instrumentation logs for these monitoring wells are provided in Appendix B of the draft RI/FS Work Plan.

Boring logs for PSARA borings SD-001 to SD-007, included the following observations:

SD-001

This boring was installed to the northeast of the Quarry Pond, just north of the access road. Soil logs indicated 6 inches of soil over 4 inches of asphalt and 8 inches of brown silty clay with brick fill material. Green to gray staining and faint hydrocarbon odor in brown silty clay with small gravel was observed at 8-10 ft-bgs. No headspace samples collected until 14-16 ft-bgs interval. Headspace readings for organic vapors included methane at 280 ppm for the 14-16 ft-bgs interval, 160 ppm for the 16-18 ft-bgs interval and 300 ppm for the 18-20 ft-bgs interval, located in sand and sand and gravel with some clay/silt. The water table was observed at a depth of 12 ft-bgs. TCE (4.5 μg/L), 1,1-DCE (0.5 μg/L), benzene (1.2 μg/L) and toluene (1.5 μg/L) were detected in the groundwater sample collected from 19 ft-bgs. Similar concentrations of these chemicals, along with 0.9 μg/L of 1,2-dichloroethane (1,2-DCA), were detected in the second groundwater sample collected at 34 ft-bgs.

SD-002

This boring was installed just north of the east-west access road, 450 feet east of the location for SD-001. Soil boring logs indicated black mottling, glass, and other debris fragments at 0-4 ft-bgs, rusty brown mottles and streaks at 12-14 ft-bgs. Headspace readings for organic vapors, including methane, were as follows: 400 ppm for the 20-22 ft-bgs interval, 180 ppm for the 22-24 ft-bgs interval and 160 ppm for the 24-26 ft-bgs interval, with the borehole completed in gravel, sand and silt and sand with clay and gravel. The water table was observed at 12 ft-bgs. Groundwater samples were collected from the 22 and 32 ft-bgs intervals. The groundwater sample collected from 22 ft-bgs contained concentrations of 1.2 μg/L 1,1-DCA, 0.9 μg/L cis-1,2-DCE, 0.8 μg/L

benzene, 1.9 μg/L toluene, and 0.5 μg/L 1,2-DCA. The 32 ft-bgs groundwater sample contained similar concentrations as the 22 ft-bgs groundwater sample, but also contained 0.9 μg/L vinyl chloride (VC).

SD-003

This boring was installed northwest of the northwest corner of the Quarry Pond. The soil boring was terminated at 6 ft-bgs due to the presence of buried waste. Headspace reading of 540 ppm [comparison of FID and photoionization detector (PID) readings indicated mostly methane] were measured in very sticky black to brown sand with black-stained silt and clay in the 2-4 ft-bgs interval. No groundwater samples were collected.

SD-004/004A

These borings were installed west of the access road, approximately 200 feet north of SD-003. The water table was observed at a depth of 12 ft-bgs. Groundwater samples were collected from 23 and 28 ft-bgs. The groundwater sample collected from 23 ft-bgs contained 1.5 μg/L of TCE, 0.9 μg/L of 1,2-DCA, 0.8 μg/L of benzene, 2.4 μg/L of toluene, 0.8 μg/L of ethylbenzene, 0.6 μg/L of 1,2,4-trimethylbenzene, 0.5 μg/L of o-xylene, and 1.2 μg/L of m,p-xylenes. The 28 ft-bgs groundwater sample contained 2/2.2 μg/L of TCE, ND (0.5)/0.8 μg/L 1,2-DCA, 0.6/0.5 μg/L benzene, 1.5/1.5 μg/L toluene and 0.7/ 0.7 μg/L m,p-xylenes.

SD-005

This boring was installed to west side of the east-west access road, approximately 50 feet west of the southwest corner of the concrete pad for the air curtain destructor (ACD). The water table was observed to be at 18 ft-bgs. Groundwater samples were collected from 28 and 43 ft-bgs. The groundwater sample collected from 28 ft-bgs contained 0.7 μg/L benzene, 2.1 μg/L toluene, 0.9 μg/L m,p-xylenes, and 0.6 μg/L ethylbenzene. The groundwater sample collected from 43 ft-bgs contained 1.6 μg/L benzene, 2.9 μg/L toluene, 0.9 μg/L m,p-xylenes, 0.7 μg/L ethylbenzene, 2.4 μg/L TCE, 0.5 μg/L of 1,2,4-trimethylbenzene, and 0.7 μg/L o-xylene.

SD-006/006A/006B

These three 2-6 foot deep borings were installed north of the northern access road and between 50 - 100 feet north-northwest of the air curtain destructor. Black slag-rich fill with cinders, ash, burnt wood fragments and debris was encountered in all three borings. SD-006 was abandoned at 4 ft-bgs due to headspace readings of 500 ppm (FID/PID comparison indicated mostly methane). Headspace readings of 1,000 ppm in the 2-4 ft-bgs interval and in the 4-6 ft-bgs interval were measured for SD-006A. SD-006B was abandoned at 2 ft-bgs due to the presence of a strong organic odor. The water table was not encountered, hence no groundwater samples were collected.

SD-007

This boring was completed 75 feet northeast of the northeast corner of the former air curtain destructor. The boring was completed at 14 ft-bgs. Soils encountered included fill containing slag, cinders, burnt wood, ash, glass and black sand down to a depth of 12 ft-bgs. The headspace readings (FID/PID comparison indicated mostly methane) were 100 ppm for the 8-10 ft-bgs interval and 300 ppm for the 10-12 ft-bgs interval. The water table was not encountered; therefore, no groundwater samples were collected.

PSARA collected one round of groundwater samples from each of these temporary wells. The groundwater analytical data, including analytical parameters, are summarized in Table 2.5 of the Work Plan. These data were consistent with the results of analyses of the groundwater samples from the temporary monitoring wells.

K-A 1.7 1996 OHIO EPA SITE TEAM EVALUATION PRIORITIZATION (STEP)

The 1996 Ohio EPA STEP investigation was completed to determine if previous disposal at the Site had impacted the environment. The STEP included the following activities:

- Review of the Site background, setting, and hydrogeology; and
- Collection of twelve soil samples (including one duplicate and one background), six sediment samples (including one duplicate), and five groundwater samples (including one duplicate and one background). Analytical results for the soil, sediment, and groundwater are summarized in Tables 2.2, 2.3, and 2.5, of the draft RI/FS Work Plan respectively.

The results for the STEP sample program is summarized below:

Soil Sample S01

This subsurface soil sample was collected 4-4.5 feet bgs in a former drum area near the north-central area of Site (south of north access road and east of central north-south access road). Analytical results indicate a concentration of 59 µg/Kg tetrachloroethene (PCE) and other chemical concentrations.

Soil Sample S10

This surficial soil sample was collected from a depth of 0 - 4 inches bgs in the area of a drum just to the south of the ACD. The sample contained 11 µg/Kg TCE, 16.3 mg/Kg cadmium, 191,000 mg/Kg copper, 12,100 mg/Kg lead, 139 mg/Kg nickel, 7.6 mg/Kg silver, 11,500 mg/Kg of zinc and other chemical concentrations.

Soil Sample S11

This soil sample was collected from 3 to 4 inches bgs in the ravine located to the west of Parcel 5175. The sample contained 10.2 mg/Kg total SVOCs, 252 mg/Kg lead, and low concentrations of other inorganic chemicals.

Soil Sample S08

This surficial soil sample was collected near drums that were found along the western slope of Parcel 5177, towards the GMR. The sample contained 16 µg/Kg methylene chloride, 12.2 mg/Kg total SVOCs, 5.4 µg/Kg Endosulfan II, and metals including, but not limited to, 14,300 mg/Kg aluminum, 278 mg/Kg antimony, 141 mg/Kg arsenic, 13,000 mg/Kg barium, 62 mg/Kg chromium, 1,830 mg/Kg copper, 59,500 mg/Kg iron, 652 mg/Kg lead, 78.1 mg/Kg nickel, 286 mg/Kg zinc, and 2.3 mg/Kg cyanide.

Soil Sample S09

This surficial soul sample was collected near drums along western slope of Parcel 5177 leading down to the GMR. The sample contained 23.7 mg/Kg total SVOCs including 18 mg/Kg butylbenzylphthalate, 830 µg/Kg Aroclor-1254 and 1,200 µg/Kg Aroclor-1260, along with metals including, but not limited to, 36 mg/Kg arsenic,

824 mg/Kg barium, 2.6 mg/Kg beryllium, 3.9 mg/Kg cadmium, 50.7 mg/Kg chromium, 1,680 mg/Kg copper, 1,990 mg/Kg lead, 85 mg/Kg nickel, 291 mg/Kg zinc, and 3.7 mg/Kg cyanide.

Sediment Sample S15

This sediment sample was collected from the northwest corner of the Quarry Pond at a depth of 15 to 18 feet below the water surface. The sample contained 0.8 μg/Kg TCE, 7.6 mg/Kg total SVOCs, 660 μg/Kg Aroclor-1254, 12 μg/Kg alpha-Chlordane, 9.6 μg/Kg Dieldrin, and 34 μg/Kg Endrin. Inorganic chemicals were also detected in this sample.

Sediment Sample S16

This sediment sample was collected in the northeast corner of the Quarry Pond at a depth of 15 to 18 feet below the water surface. The sample contained 21.1 mg/Kg total SVOCs and 545 mg/Kg manganese and other inorganic chemicals.

Sediment Sample S17

This sediment sample was collected off of the bank of the GMR approximately 350 ft west of the center of the Site (center of Parcel 5177). The sample contained 0.7 μg/Kg TCE, 23.1 mg/Kg total SVOCs, and 0.65 mg/Kg mercury and other inorganic chemicals.

Sediment Sample S18

This sediment sample was collected from the GMR at a location downstream of the Site. The sample contained 9.1 mg/Kg total SVOCs and inorganic chemicals.

Sediment Sample S19

This sediment sample was collected in the GMR, 350 feet west of the former auto salvage yard (west of north part of Lot 5177) and contained 17.6 mg/Kg SVOCs and inorganic chemicals.

Groundwater Analytical Results:

Analytical results for MW-101 indicate chlorinated solvents including 13 µg/L 1,1-DCA, 150 µg/L 1,2-DCE (total) and 4 µg/L VC. The groundwater sample from MW-102 contained 22 µg/L chloroethane, 15 µg/L toluene and 4 µg/L xylenes. The groundwater sample from MW-104, located on DP&L property (used as a background well) contained 547 µg/L arsenic.

Ohio EPA used a criterion of three times the background concentration [characterized by the collection and analysis of one soil sample (S07, southwest end of Quarry Pond) and one groundwater sample (MW104, east of the Site)] to determine if constituents detected in soil, sediment, and groundwater were of concern. Based on this evaluation, the Ohio EPA concluded the following constituents were present in the samples collected at concentrations that were elevated above background.

Parameter	*Soil*	*Sediment*	*Groundwater*
Chlorinated VOCs	×		×
Acetone			×
Toluene			×
PAHs	×		
Phthalates	×		
Pesticides		×	×
Metals	×	×	× (potassium)
PCBs	×	×	

Note:
× = detected at a concentration at least three times above background concentration

There was no statistical evaluation of background soil and water quality, so this evaluation is somewhat qualitative.

The background sediment sample OEPA used for the sediment evaluation (S19) was collected west of the Site, west of Valley Asphalt, and is not actually representative of background concentrations.

Exposure Pathways

The STEP Report concluded that the human health soil exposure pathway was determined to be potentially complete at the Site due to a lack of access control, such as a fence.

The STEP Report also concluded that the groundwater exposure pathway was potentially complete. The uncertainty associated with this pathway was due to the undefined groundwater flow direction and the presence of other sources of groundwater contamination in the area.

With respect to surface water and sediments, the exposure pathway was determined to be potentially complete due to the detections in sediment samples.

The STEP report concluded that the presence of soil and debris piles, along with the 1996 PSARA data, resulted in a potentially complete air exposure pathway. The STEP Report identified that the presence of PAHs in some of the samples could be attributed to the Valley Asphalt plant. However, Alcine Grillot and soil boring logs also indicate asphalt was disposed at the Site.

K-A 1.8 1996 TO 2005 PFI SITE INVESTIGATIONS

Based on a review of the available Site history, the Site setting, and previous investigations conducted by others, PFI completed a series of investigations on behalf of Coolidge, Wall, Womsley & Lombard (representing some of the Site property owners), to aid in defining the environmental issues at the Site.

PFI supervised the drilling of thirteen soil borings at the Site in 1998 and 1999. PFI completed ten of the borings as 2-inch PVC groundwater monitoring wells (MW201-204, MW206-210, and MW212), and one of the borings was completed as a piezometer (P211). The boring logs indicate P-211 was constructed in the same way as the groundwater monitoring wells (i.e., using 2-inch PVC). However, it appears that P-211 was only monitored for groundwater elevation measurements, not analytical parameters. The two remaining soil borings (GT-205 and GT-212) were not completed as monitoring wells due to the presence of heaving sands in the well completion interval.

PFI installed surface water elevation gauges in May 1998 at the Quarry Pond, Large Pond, and Small Pond. PFI used these gauges to monitor surface water elevations in 1998 and 1999, in connection with the groundwater elevation measurements, which were collected approximately quarterly from June 1998 through August 2005.

PFI collected up to 10 rounds of groundwater samples and analyzed the samples for the parameters indicated below. Note that the TCL list was not utilized. 1,2,4-Trimethylybenzene, cis-1,2-DCE, ethylbenzene, and o-xylene were not included in the analyses. The analyses included total xylenes.

Monitoring Round Date	*Parameters Analyzed*
• January 1998	VOCs, Resource Conservation and Recovery Act (RCRA) Metals
• May 1998	VOCs, RCRA Metals, Natural Attenuation Indicators [2]
• February 1999	VOCs, RCRA Metals, Natural Attenuation Indicators
• November 1999	VOCs
• May 2000	VOCs
• June 2001	VOCs
• June 2002	VOCs
• July 2004	VOCs
• October 2004	VOCs
• August 2005	VOCs

The groundwater analytical data are presented in Table 2.5 of the draft RI/FS Work Plan along with Ohio EPA's and PSARA's data. Table 2.5 of the Work Plan also depicts those parameters that were not analyzed for in a given sample collection round.

PFI sampled surface water and sediments at the Quarry Pond during April 1999 and May 2000. PFI collected three surface water samples during each sampling event using a Bacon Bomb sampler, and three sediment samples during each event using an Ekman

[2] Chloride; Nitrate; Ammonia as Nitrogen; Sulfate; Total Alkalinity; Total Organic Carbon; Methane; Ethane; Ethene; and Dissolved Iron

Dredge. PFI analyzed the samples for VOCs and also analyzed the April 1999 sediment samples for total organic carbon (TOC). The surface water analytical data are presented in Table 2.7 of the Work Plan. The sediment analytical data are presented in Table 2.3 of the draft RI/FS Work Plan.

K-A 1.9 SUMMARY OF RESULTS OF PFI INVESTIGATION

Based on the PFI results, groundwater quality at the Site has been impacted by chlorinated solvents, and inorganic chemicals including, but not limited to, arsenic and lead. In particular, TCE has been detected consistently in groundwater samples from wells completed on the eastern (MW-202 and MW-210) and western (MW-201[3]) boundaries of the Site. TCE has also been detected on occasion in groundwater samples from MW-102 and MW-208, also located at the western and eastern margins of the Site, respectively.

PFI noted that breakdown products from the degradation of TCE (1,2-DCE and VC) have been consistently detected in groundwater samples collected from MW-101A (south-central portion of the Site). 1,2-DCE has also been consistently detected in groundwater samples collected from MW-210 at the southeast corner and once in groundwater samples from MW-202 on the eastern margin of the Site. 1,2-DCE and VC have been detected on occasion in groundwater samples from MW-203 and MW-208 at the southern and eastern margins of the Site, respectively. However, as noted by USEPA, the presence of these "daughter" compounds could be attributed to co-solvent deposition rather than degradation.

In addition, PFI also noted that 1,1,1-TCA and its potential breakdown products have been detected in groundwater samples collected from monitoring wells installed at the Site. The presence of both parent and daughter compounds may indicate that natural attenuation is occurring at the Site. As noted above, the mere presence of these compounds does not definitively mean that biodegradation is occurring or that biodegradation and natural attenuation are effective remedial processes. Investigative activities would be needed to evaluate this line of evidence further.

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3 Although groundwater samples collected from MW-103 in the late 1990s contained low concentrations of TCE, TCE has not been detected in groundwater samples collected from this well from 2000 and later.

PFI also collected sediment and surface water samples from the Quarry Pond. These data are presented in Tables 2.3 and 2.7, of the draft RI/FS Work Plan respectively. PFI noted that two of the three sediment samples contained TOC (although the presence of TOC may or may not be evidence of impact) and none of the surface water or sediment samples contained detectable concentrations of VOCs.

Notwithstanding the above discussion, PFI noted that seasonal fluctuations in water table depth can cause variations in groundwater flow direction(s) and hence may affect groundwater quality at a given monitoring well location. Repeated sampling events, scheduled to coincide with the variations in flow direction, would be required to confirm the reduction in concentration of chlorinated VOCs is not related to seasonal flow direction variation.

K-A 1.10 SUMMARY - 2000 TCA ENVIRONMENTAL REPORT - VALLEY ASPHALT

As was discussed in Section 2.7.1, Valley Asphalt retained TCA to oversee the removal of contaminated soil and drummed waste identified on the Valley Asphalt property. Analytical results for the composite waste sample collected include:

- 75 mg/Kg Aroclor 1254;
- 7 mg/Kg benzene;
- 2.5 mg/Kg 2-butanone;
- 1.7 mg/Kg chlorobenzene;
- 84 mg/Kg ethylbenzene;
- 18 mg/Kg 4-methyl-2-pentanone;
- 530 mg/Kg toluene;
- 64 mg/Kg TCE; and
- 340 mg/Kg xylenes.

It appears that five drums containing a solid material were removed, characterized as a characteristic hazardous waste (lead and cadmium) with PCBs, and disposed of at the Clean Harbors facility in Cincinnati, Ohio. A total of 2,217 tons of non-hazardous impacted soil containing VOCs was disposed at Waste Management's Stony Hollow Landfill in Dayton, Ohio.

TCA identified a drinking water well and production well located in the vicinity of the excavation area. TCA collected groundwater samples from these wells. No VOCs were detected in the samples collected from either well. The TCA report did not indicate whether the wells were subsequently abandoned.

The TCA report does not describe the condition of the excavation prior to being backfilled. However, CRA spoke with Dale Farmer, Ohio EPA's On Scene Coordinator on December 15, 2006 who advised that the drums encountered had been crushed prior to being excavated, and that there was a corner of a drum and other debris visible in the side wall of the excavation. The excavation was backfilled without any further investigation conducted. Mr. Farmer stated that no intact drums or complete drum carcasses were excavated nor were any complete drum carcasses observed in the side walls of the excavation.

In January 2006, Ohio EPA visited the Valley Asphalt property to determine the status of the two water wells that were reported by TCA in their 2000 Environmental Report. The report stated that TCA sampled the wells, but did not detect any VOCs in the water samples. One of these two wells was identified on a sketch in the TCA report. This well, situated approximately 50 feet southwest of the drum excavation, was located by Ohio EPA on January 20, 2006, next to what appears to be a truck-wash area. Its location suggests it is potentially down gradient of the 2000 excavation. Ohio EPA meeting notes with TCA dated May 31, 2000 state that this well was used minimally for sanitary purposes; however, during reconnaissance on January 20, 2006, Mr. Hutch Rogge, project manager of John R. Jurgensen Co. (owner of Valley Asphalt), stated that he thought the well provided drinking water to the main office.

Upon inspecting the well, Ohio EPA noted that the well lacked a protective cover or sealing cap. The well casing was covered with a plastic bag. A large diameter concrete pipe surrounded the protective casing. The annular space was filled with trash, including a spray can. The employees were not familiar with any other wells located on the property.

APPENDIX K-B

TEST PIT/TEST TRENCH INVESTIGATION LETTER

CONESTOGA-ROVERS
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May 9, 2008

Reference No. 038443

Karen Cibulskis
Remedial Project Manager
United States Environmental Protection Agency
Region V
77 West Jackson Boulevard
Mail Code SR-6J
Chicago, IL 60604

Dear Karen:

Re: Final Test Pit/Test Trench Investigation
South Dayton Dump and Landfill Site, Moraine, Ohio (Site)

This Letter Work Plan presents the scope of work for a test pit and test trench investigation of parts of the Site. Conestoga-Rovers & Associates (CRA) has prepared this Letter Work Plan on behalf of the South Dayton Dump and Landfill Potentially Responsible Party Group (PRP Group).

This Letter Work Plan is based on the February 12 and 27, 2008 discussions between the PRP Group and United States Environmental Protection Agency (USEPA) regarding the additional data that the PRP Group would like to collect for the Feasibility Study (FS). The Letter Work Plan also incorporates comments from the USEPA on a draft that was discussed at the February 27, 2008 meeting. The Letter Work Plan incorporates comments received from USEPA on April 15, 2008.

The objectives of the test pit and test trench excavation and sampling are as follows:

- collect data to assist in identifying the nature and delineating the extent of various types of landfilled materials above the water table;
- collect data to assist in characterizing landfill materials above the water table;
- collect data to assist in characterizing leachate from unsaturated landfilled material;
- assess areas of the Site previously identified as specific areas of concern [i.e., Valley Asphalt drum removal area, Valley Asphalt former underground storage tank (UST) area (a.k.a. Dayton Recycling), Custom Delivery UST area, Lot 4423, etc.); and
- identify Site areas, which may require further investigation (for example leachate sampling and analysis, groundwater quality investigation, or other delineation work).

The test pit and test trench investigations will be completed after the Land Survey, Bathymetry Survey, and Geophysical Investigation, and Leachate Seep Investigation have been completed. A schedule, including a Gantt chart, for the investigative activities to be completed at the Site in 2008 was provided to USEPA on March 11, 2008. The locations of the test pits and test trenches may be adjusted based on the results of these previously mentioned investigations and upon consultation with the USEPA.

TEST PITS/TEST TRENCHES

Test pits and test trenches are proposed in locations where the PRP Group would like to collect additional information about the depth and nature of the fill material above the water table. The information will be used to verify the limits of fill and to assist in characterizing the nature of the landfilled materials present in the areas investigated.

Six test pits will be excavated in the central portion of the Site. Twenty-three test trenches will be excavated throughout the Site.

The locations of the test pits and test trenches will be finalized based on the results of the geophysical investigation (the USEPA may be asked to approve moving, relocating, or adding test pit and test trench locations based on field observations, geophysical investigation results, etc.). The nature and depth of fill material above the water table will be visually identified and recorded. Test trenching will focus on the perimeter of the PRP Group's preliminary direct contact presumptive remedy area, which was defined in the Remedial Investigation/Feasibility Study (RI/FS) Statement of Work (SOW) and the area immediately beyond the perimeter. In addition, the test trenching will assist in identifying and characterizing fill material at locations along the western embankment of the Site. Excavations will be completed to the depth of the water table, where possible (as limited by the ability of the excavator to reach the depth of the water table, the stability of the walls of the excavation, and/or the presence of obstructions). If an obstruction is encountered during the excavation of a test trench, the location of the trench will be adjusted to avoid the obstruction. If excavation to the water table is not possible due to the depth of the water table or the stability of the fill material, the PRP Group will consider the need for additional investigation at the location in question during future investigation work. The potential impacts from saturated fill materials will be assessed as part of the groundwater investigation proposed for the Site (under separate cover). The utility of this information to the FS is discussed above.

Test pits and test trenches will be excavated in the locations shown on Figure 1. As noted above, the locations of the test pits and test trenches may be adjusted based on the results of the

Land Survey, Bathymetry Survey, and Geophysical Investigation, and the Leachate Seep Investigation and upon consultation with the USEPA. Each test pit will be approximately 6 feet long by 3 feet wide and will extend to the water table, if the excavation can be completed safely to that depth (i.e., stable slopes and excavation sidewalls, no buried structures, etc.) and the excavator is capable of reaching that depth.

Each test trench will be approximately 30 feet long by 3 feet wide, and will extend to the water table (if this can be excavated safely) and horizontally to the visual limit of fill. If the horizontal limit of fill is not determined in any planned 30-foot trench, to the extent practical (i.e., where not impeded by the presence of surface structures, property boundaries, unstable slopes or side walls, buried structures, etc.), the test trench lengths will be extended to attempt to visually locate the edge of the fill. This visual limit (both lateral and vertical) will be determined by the presence of undisturbed native soil in the excavation. CRA will also note if fill material appears to consist of re-located spoils from the gravel extraction operation versus undisturbed native material; however, the presence of relocated spoils will not be used as an indicator that other wastes have not been disposed at an individual location. Test trench excavation will continue in these areas to the depth of native material or the maximum reach of the excavator, whichever is less.

The nature and depth of fill material will be visually identified and recorded. The procedures and equipment to be used to excavate trenches and visually characterize the fill material are described below.

TEST PIT AND TEST TRENCH EXCAVATION PROCEDURES

An excavator or extended reach backhoe will be used to excavate the test pits and test trenches. The reach of the excavator or backhoe will be at least 18 feet. Data regarding conditions at depths greater than those that can be reached by the excavator may be obtained during vertical aquifer sampling and monitoring well installation. The PRP Group will provide the details of any soil sampling during VAS and any revisions to the Field Sampling Plan and Quality Assurance Project Plan to EPA for review and approval prior to conducting this work. The PRP Group will also submit any revisions to the Health and Safety Plan (HASP) to EPA for review prior to conducting this work.

The test pit excavation procedures are as follows:

1. Each test pit will be assigned a unique identification number. Prior to starting the test pit excavations, the locations of each test pit and test trench will be staked in the field

using the locations identified on Figure 1. As noted above, the locations of the test pits may be adjusted based on the results of the Land Survey, Bathymetry Survey, and Geophysical Investigation, and the Leachate Seep Investigation and upon consultation with the USEPA;

2. The area immediately adjacent to the test pit will be covered with two layers of 6-mil polyethylene sheeting for stockpiling excavated fill material. The polyethylene sheeting and excavation spoils will be placed downwind of field personnel and in such a manner that water runoff from the fill material will be directed back into the excavation. If possible, fill material temporarily stockpiled on the liners will be backfilled into the open excavations before the contractor leaves the Site for the day. If the fill material cannot be backfilled at the end of the workday, the contractor will ensure the material is covered securely with a polyethylene liner to control potential emissions and to minimize the exposure of the material to rainwater. The contractor will also ensure that temporary fencing is placed around the stockpiled material and the open excavation;

3. The test pits will be approximately 3 feet wide and will extend to the depth of the water table, where possible and feasible (as limited by the ability of the excavator to reach that depth, the stability of the walls of the excavation, and/or the presence of obstructions). The lengths of individual test pits will be determined in the field by the field representative based on conditions encountered during excavation. If obstructions are encountered and sidewalls are stable, then the width or length of the test pit may be expanded to aid in excavating to depth. Excavation at each location will be completed in a controlled manner so as to minimize damage to any potentially intact drums. If a test pit cannot be excavated to the surface of the water table due to obstructions or sidewall instability, and the excavation equipment is capable of reaching that depth, the test pit will be relocated 50 feet (or a lesser distance if appropriate) from the original location and attempted again. If, during the excavation of a test pit, PID, particulate, or vinyl chloride readings above the action levels in the HASP are recorded, excavation of the test pit will cease and the Site Supervisor (SS) will evaluate what actions (i.e., upgrade in level of personal protection equipment or termination and backfilling of test pit) are appropriate. If during the excavation of a test pit, combustible gas, oxygen, hydrogen sulfide, carbon monoxide, or radiation readings exceed (or in the case of oxygen fall below) an action level, the test pit excavation will be immediately stopped and the test pit backfilled, provided it is safe to do so. The test pit will be relocated 50 feet (or a lesser distance if appropriate) from the original location and attempted again. The location will be documented, and, if appropriate and safe to do so, may be investigated further during other investigative activities at the Site (i.e., Groundwater Investigation, Landfill Gas/Soil Vapor Investigation, etc.);

4. CRA will observe the materials excavated and record the nature of the materials on a test pit stratigraphy log. The test pits will be excavated in two to three foot increments to aid in accurately determining the depth of discrete layers of fill material and the fill material/native material interface. Where appropriate, and where it is safe to do so, CRA will measure the depth of the test pit excavation where specific layers of fill material are encountered and the total depth of the excavation. The observations will include a visual description of the types of material (i.e., undisturbed native soil, spoil from quarry operations, domestic refuse, industrial refuse, metallic debris, ash, fly ash, construction and demolition debris, foundry sand, asphalt, slag, or other appropriate description) and, if possible, a Unified Soil Classification System (USCS) description. Soils will be logged using the USCS by an on-Site geologist. Soil classification methods will include visual assessment, texture assessment, dry strength tests, toughness tests, and dilatancy tests, as appropriate depending on the nature of the soil encountered. The visual classification of waste materials is, by its very nature, somewhat arbitrary. The on-Site geologist will be experienced in performing such observations, which will be based on the physical nature of the material encountered. As detailed below, samples of distinct fill materials will be retained in the event that the classification of specific materials needs to be revisited in future. Photographs of the material will also be included;

5. Empty drum overpacks will be maintained at the Site during excavation. Should an intact waste container be damaged during excavation the drum management procedures presented in Attachment A will be implemented; and

6. Each test pit will be backfilled with the excavated materials in reverse order to that in which they were removed. The test pits will be restored to match surface conditions prior to excavation. During backfilling of the test pit, the bucket of the excavator will be used to compact the material as it is placed in the excavation in order to ensure that any expansion of the materials that occurs during excavation is reversed and the test pits can be restored to grade.

Access of the general public and on-Site commercial/industrial workers to the investigative locations will be restricted by the SS and air monitoring will be used to ensure that any emissions generated during test pitting activities do not pose a risk to the general public or on-Site workers. On-Site commercial/industrial workers will be notified in advance of intrusive activities that may have the potential to generate emissions, where these intrusive activities are located proximally to an active on-Site commercial/industrial facility.

Test trenches will be excavated in the same manner as detailed above for test pits except that test trenches will be excavated to the top of the water table in a continuous length of approximately 30 feet or the horizontal limit of fill (if undisturbed native soil is encountered before reaching 30 feet) as discussed above.

To the extent possible given the available data for the Site, CRA will attempt to start the excavation in areas of fill (i.e., non-native) material and continue the excavation towards the presumed location of native material. If fill is encountered at the start of the trench, the trench will be excavated in the presumed direction of native material, e.g., away from the PRP Group's direct contact presumptive remedy area. If native material is encountered at the start of the trench, the trench will be excavated in the presumed direction of fill material, e.g., towards the PRP Group's direct contact presumptive remedy area. As noted above, if the horizontal limit of fill is not determined in any planned 30-foot trench, to the extent practical (i.e., where not impeded by the presence of surface structures, property boundaries, unstable slopes or side walls, buried structures, etc.), the test trench lengths will be extended to attempt to visually locate the edge of the fill. Where further extension of a test trench is not feasible and/or practicable, the PRP Group may, in consultation with the USEPA Site representative(s), elect to abandon a test trench location and install an additional test trench off-set from the original location in the presumed direction of the native/fill material, as appropriate. As noted above, the locations of the test trenches may be adjusted based on the results of the Land Survey, Bathymetry Survey, and Geophysical Investigation, and the Leachate Seep Investigation and upon consultation with the USEPA.

If clean backfill material is encountered during any of the test trenches proposed in the Valley Asphalt drum removal area, the Dayton Recycling UST removal area, or the Custom Deliveries UST removal area, CRA will attempt to continue the test trench excavation away from the location of the clean backfill material or relocate the test trench outside the clean backfill material, as appropriate depending on the size of the original excavation.

The test trenches will be used to visually determine the limits of the fill and to provide information on the nature of the fill material at these locations.

TEST PIT AND TEST TRENCH SAMPLING

The following sampling procedures and associated tasks will be performed as part of the Test Pit/Test Trench Investigation:

1. CRA will prepare a photographic log of each test pit excavation during its progression. The photographic record will list the date of each photograph, a specific description of what the photograph depicts, its location, and the photographer;
2. The dimensions of each excavation and a description of the materials encountered during excavation will be recorded on a Test Pit Stratigraphy log, an example of which is contained in Attachment B;
3. Samples of the fill will be collected, from each sidewall and the base of the excavation during the excavation. A minimum of one sample collected from each test pit and two samples collected from each test trench will be submitted to an analytical laboratory for analyses. The specific material selected for sampling and the number of samples will be determined in the field by the CRA field representative and reviewed with the USEPA Site representative(s). Sample selection will be based on the visual appearance of the material (for example, color, staining, grain size, etc.), location of the material prior to removal (for example, adjacent to drums or base of excavation), and field instrument measurements [i.e., headspace readings using a photo-ionization detector (PID)]. CRA will collect a sample of each visually distinct layer of fill type for headspace analysis. Where fill material encountered is not visually distinct with depth, CRA will use visual and olfactory evidence of contamination and PID screening of the soil as it is excavated to identify appropriate samples for headspace screening. All olfactory evidence will be obtained taking care to limit exposure to any vapors and in accordance with the HASP. At a minimum, if fill material is not visually distinct with depth, samples will be collected for headspace screening approximately every five feet vertically. The headspace analysis will aid in the selection of the discrete samples to be analyzed from each excavation and in the selection of the sample(s) to be retained from each distinct fill type based on visual observations and headspace analysis (see below). The observations will be recorded in the Test Pit Stratigraphy log. The samples will be collected directly from the bucket of the excavator and/or the stockpiled spoils. The sample collection procedures are identified in the Field Sampling Plan. Fill material samples will be collected in an attempt to characterize distinct fill zones or landfilled materials based on visual observations, PID readings, and the analytical data generated from the program as discussed below. CRA will also use representative fill samples retained (see below) from each test pit and test trench to compare fill types from different excavations. Samples of the same distinct fill zones or landfilled materials

based on visual observations and headspace analysis will be collected from multiple test pit and test trenches where possible, i.e., where the same distinct fill zone or landfilled materials based on visual observations and headspace analysis are present in more than one test pit in recoverable quantities;

4. A portion of each sample will be placed in a separate container for headspace analysis using a PID. Results of the headspace analysis will be recorded in the Test Pit Stratigraphy log. A sample from each distinct fill type observed in each test pit and test trench will be retained in appropriate sampling containers maintained at appropriate temperatures so that samples may be submitted in the future (within the applicable sample holding time) for laboratory analysis. Field observations and field screening results will be reviewed with the USEPA's Site representative(s) on a daily basis;

5. Daily proposed sample submissions to the analytical laboratory will be reviewed with the USEPA's Site representative(s). At a minimum, samples of each distinct fill type (based on visual observations and headspace analysis) encountered at the Site will be submitted for the following analyses: Target Compound List (TCL) volatile organic compounds (VOCs), TCL semi-volatile organic compounds (SVOCs), TCL herbicides and pesticides, TCL polychlorinated biphenyls (PCBs), and Target Analyte List (TAL) inorganics. Where field observations and field screening indicate that similar types of fill material in different test pits/test trenches may be from different sources (e.g., visually similar materials in two distinct and separate layers within a trench or at widely varying depths within adjacent trenches, or visually similar materials in different trenches in different areas of the Site), additional samples may be submitted. Additional samples may also be submitted where visually similar fill materials are potentially impacted by different contaminants (e.g. visually similar materials where one has a strong odor and the other a high organic vapor content as measured using a PID).

   Multiple samples of similar fill types based on visual observations and headspace readings encountered across the Site will be submitted for TCL/TAL laboratory analysis to assess the variability of the analyzed materials within the Site. Ash fill materials encountered will be collected and submitted for dioxin and furan analyses. Up to 10 samples of ash will be submitted for dioxin and furan analyses if ash is encountered in at least 10 separate excavations. If potential friable asbestos-containing materials (ACM) (i.e., ceiling tiles, wall board, pipe insulation, automotive brake pad manufacturing refuse, etc.) are encountered, a minimum of one sample of each distinct type of potential ACM will be submitted for asbestos analysis. A sampling summary is presented in Table 1. The HASP includes provisions to assess worker exposure to potentially radioactive foundry sands.

6. Should leachate seeps be identified in any of the test pits or test trenches, samples will be collected. For shallow leachate seeps that can be reached by hand from the edge of the test pit or trench, the area located immediately beneath the seep will be dug out using a clean shovel or trowel. A clean sample jar or pail will be set into the dug out area and the liquid will be allowed to gently accumulate in the container. If the depth of the excavation prohibits field personnel from safely conducting the liquid collection, sufficient saturated material in and around the seep will be excavated and placed on a polyethylene sheet and the liquid allowed to gently drain into a container. A field blank sample of distilled deionized water poured onto clean polyethylene sheeting will also be collected. The liquid will be transferred to sample containers for submission to the analytical laboratory. As the volume of liquid may be limited, prioritization of requested analyses for the sample will be as follows: TCL VOCs, TCL SVOCs, TCL herbicides and pesticides, TCL PCBs, and TAL inorganics. A sampling summary is presented in Table 1. Sampling of leachate seeps identified during the Test Pit/Test Trench Investigation will be performed in accordance with the Leachate Seep Investigation Work Plan and the Field Sampling Plan (FSP);

7. A composite sample of each fill type (i.e., construction and demolition debris, ash, and cinders, etc.) will be prepared from the retained samples of the fill types from the test pits and test trenches and submitted to the analytical laboratory for Toxicity Characteristic Leaching Procedure (TCLP) preparation with subsequent analysis of the resultant leachate for VOCs, SVOCs, herbicides, pesticides, and metals. Samples will also be analyzed for PCBs, corrosivity, ignitability, and reactive cyanide and sulfide. A minimum of one composite sample will be submitted for each fill type. Where similar fill types are present in widely separated locations, additional samples may be submitted. The parameters and associated analytical methods are specified in Table 1; and

8. Duplicate photographs and the corresponding photographic record will be provided to USEPA and the Ohio Environmental Protection Agency (Ohio EPA) at the completion of this investigation.

The following protocol will be used to determine the number of samples to be submitted for laboratory analysis. Specific samples to be submitted for laboratory chemical analysis will be selected by the CRA field representative and reviewed with the USEPA's Site representative(s) on a daily basis. Depending on the nature of materials encountered in an individual test pit or trench, the number of samples for each medium may vary. For example, if no drums or only minimal amounts of drum remnants are observed in a test pit, samples of drum contents would not be collected. In addition, the number of samples submitted for laboratory chemical analysis

may increase or decrease depending on headspace results, field observations, the spatial distribution and types of existing data, and the number and types of samples collected.

The intent of the test pit and test trench investigation is to identify locations that exhibit similar characteristics (i.e., visual, physical, and, to the extent the materials are analyzed, chemical composition). Test pits may be grouped together based on similar field observations. Where grouping occurs, CRA will select samples from the entire grouping for chemical analysis. The CRA field representative will establish the groupings, identify which test pits and test trenches will compose a given grouping, and select fill samples for submission to the analytical laboratory for analysis. Fill materials will only be grouped together where the fill materials are present in the same area of the Site and where laboratory holding times allow. Inherent in the grouping of fill types is the presumption that analytical data and other results obtained will be representative of the entire grouping. CRA will attempt to evaluate this presumption through replicate sampling in wide spread waste types at a frequency of one replicate sample for every five grouped samples. The test pit and test trench locations that are grouped together along with the corresponding sample identification number(s) will be identified in the Test Pit Stratigraphy log. Sample selection will be performed such that fill types from multiple different locations are selected.

All work will be performed in accordance with the FSP, Quality Assurance Project Plan (QAPP), and HASP pending USEPA's approval of the relevant sections of these documents.

SCHEDULE

The test pit and test trench investigation work will commence within two weeks of the submission of the Survey and Geophysical Survey Report to the USEPA. Field activities will be completed within three weeks. CRA plans to use a single excavator to complete the test pit/test trenching activities; however, a second excavator and field crew will be added if scheduling constraints so dictate. The PRP Group will provide the USEPA with verbal notification of field activities and the number of excavators to be used at least 15 days in advance of the initiation of field activities.

REPORTING

Results of the test pit and test trench investigation will be summarized and presented in a report. The report, which will include a description of the field work completed, any deviations from this Letter Work Plan and the rationale behind the change, photographs, logs, analytical

summary tables, and analytical data reports, will be provided to the USEPA and Ohio EPA within one month of the receipt of analytical data from the laboratory. Monthly progress reports during the Test Pit/Test Trench Investigation fieldwork will include the information required for monthly progress reports in the RI/FS SOW (including test pit/test trench locations, headspace readings, visual fill descriptions, stratigraphic information, samples collected, and analytical data).

Should you have any questions on the above, please do not hesitate to contact us.

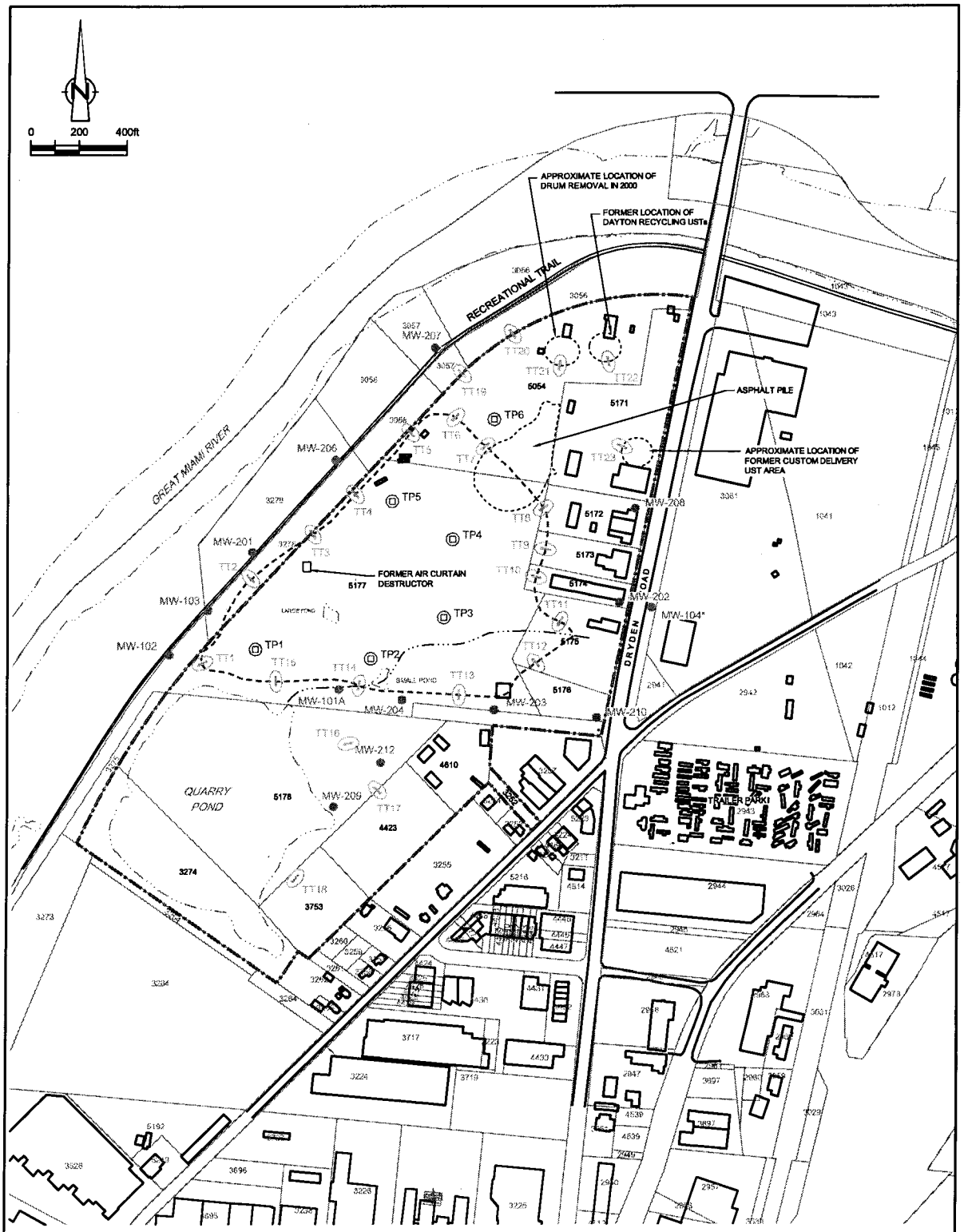
Yours truly,

CONESTOGA-ROVERS & ASSOCIATES

Stephen M. Quigley

AL/ca/23
Encl.

c.c. Matt Mankowski, USEPA (PDF)
Matt Justice, Ohio EPA (PDF)
Eric Kroger, CH2M Hill (PDF)
Scott Blackhurst, Kelsey Hayes Company (PDF)
Wray Blattner, Thompson Hine (PDF)
Ken Brown, ITW (PDF)
Jim Campbell, Engineering Management Inc. (PDF)
Tim Hoffman, Representing Kathryn Boesch and Margaret Grillot (PDF)
Paul Jack, Castle Bay (PDF)
Robin Lunn, Mayer Brown (PDF)
Roger McCready, NCR (PDF)
Karen Mignone, Pepe & Hazard (PDF)
Adam Loney, CRA (PDF)



LEGEND

MW-206 UPPER AQUIFER MONITORING WELL LOCATION

SITE BOUNDARY (SOW 2006)

PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA

TT1 PROPOSED TEST TRENCH LOCATION

TP1 PROPOSED TEST PIT LOCATION

EDGE OF WATER

APPROXIMATE LOCATION

figure 1

TEST PIT AND TRENCH EXCAVATION LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
USGS AERIAL PHOTOGRAPH, DAYTON SOUTH, 1994.

38443-00(CIBU023)GN-WA002 APR 22/2008

TABLE 1

SUMMARY OF TEST PIT/TEST TRENCH SAMPLING AND ANALYSIS PROGRAM
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Task/Event	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Sample Locations*	*Investigative Samples* [5]	*Quality Control Samples* [1] *Field Blanks* [2]	*Field Duplicates*	*MS/MSD LCS/LCD*	*Total* [3]
Test Pit Sampling	Solid	PID Screen / Visual Observation of Distinct Fill Types/Intervals	TCL VOCs, TCL SVOCs, TAL Inorganics [4], TCL Herbicides and Pesticides, TCL PCBs, TCLP [6]	6	6	1	1	1	9
Test Trench Sampling	Solid	PID Screen / Visual Observation of Distinct Fill Types/Intervals	TCL VOCs, TCL SVOCs, TAL Inorganics TCL Herbicides and Pesticides, TCL PCBs, TCLP [6]	23	46	5	3	3	57
Ash Fill Materials	Solid	Visual	Dioxins & Furans	TBD	TBD	TBD	TBD	TBD	TBD
Potential Asbestos Containing Materials	Solid	Visual	Asbestos	TBD	TBD	TBD	TBD	TBD	TBD
Leachate Sampling	Liquid	PID Screen	TCL VOCs, TCL SVOCs, TCL Herbicides and Pesticides, TCL PCBs, TAL Inorganics	TBD	TBD	TBD	TBD	TBD	TBD
Waste and/or Drum Characterization	Solid/Water	PID Screen	TCLP VOCs, TCLP SVOCs, TCLP Herbicides, TCLP Pesticides, TCLP Metals, PCBs, Corrosivity, Ignitibility, Reactive Cyanide, Reactive Sulfide	TBD	TBD	--	--	--	TBD

Notes:

1 Quality control samples will include laboratory supplied trip blank samples for volatile sample analysis with each shipping cooler of aqueous investigative samples.
2 Field blank samples consisting of equipment rinsate blanks will not be collected when dedicated or disposable sampling equipment is employed.
3 The total quantity is dependent on the actual quantity of samples and field quality control samples collected.
4 TAL Inorganics include the 23 metals and total cyanide.
5 Refers to the minimum number of investigative samples to be collected.
6 TCLP analysis will be completed on selected composite samples for the parameters listed under Waste and/or Drum Characterization as per the Letter Work Plan.

TCL - Target Compound List
VOC - Volatile Organic Compounds
SVOC - Semi-volatile Organic Compounds
TAL - Target Analyte List
PCB - Polychlorinated Biphenyls
TCLP - Toxic Characteristics Leachate Procedure
DO - Dissolve Oxygen
ORP - Oxygen Reduction Potential

ATTACHMENT A

DRUM MANAGEMENT PROCEDURES

ATTACHMENT A

DRUM MANAGEMENT PROCEDURES

The following presents the procedures associated with drum identification, management and sampling:

- Markings on any drums or other waste containers encountered will be examined, documented, and photographed and keyed to a unique drum identification number;
- The contents of a representative number of drums or other waste containers encountered will be sampled. The containers to be sampled will be selected by the field representative. Samples will be collected in or near test pits from containers that are ruptured and whose contents are readily accessible. Samples from undamaged drums will be collected from the drum following placement in the overpack. Liquid samples will be analyzed for the parameters and using the methods specified in Table 1; and
- Empty drum overpacks will be maintained at the Site during excavation. Should an intact waste container be damaged during excavation, it will be immediately removed from the excavation and placed in an overpack. Any material that becomes visibly impacted by a release from a damaged waste container will also be removed from the excavation and placed on a separate sheet of polyethylene adjacent to the test pit. All overpack drums and excavated visibly impacted material will be handled in accordance with the procedures detailed in the Field Sampling Plan for handling investigation-derived wastes.

ATTACHMENT B

TEST PIT STRATIGRAPHY LOG

TEST PIT STRATIGRAPHY LOG

Page ___ of ___

Project Name:	**Contractor:**	**Test Pit Designation:**
Project Number:		**Date Started:**
Client:	**Surface Elevation:**	**Date Completed:**
Location:	**Test Pit Method:**	**CRA Supervisor:**

Depth (m/ft)			*Soil Symbol, Primary Component, Secondary Components, Relative Density/Consistency, Grain Size/Plasticity, Gradation/Structure, Colour, Moisture Content, Supplementary Descriptors*	*Sample No.*	*Sample Interval*	*PID*	*Location:*
From	*At*	*To*					*Geologic Profile*

Completed by: _____

Date: _____

CRA

APPENDIX K-C

GROUNDWATER LETTER WORK PLAN

CONESTOGA-ROVERS
& ASSOCIATES

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May 7, 2008

Reference No. 038443

Ms. Karen Cibulskis
Remedial Project Manager
United States Environmental Protection Agency
Region V
77 West Jackson Boulevard
Mail Code SR-6J
Chicago, IL 60604

Dear Karen:

Re: Final Groundwater Letter Work Plan
South Dayton Dump and Landfill Site Moraine, Ohio (Site)

This Letter Work Plan presents the South Dayton Dump and Landfill Potentially Responsible Party Group's (PRP Group's) approach for investigation of subsurface and groundwater conditions at the Site. The work will help address data gaps and provide information to aid in the completion of a Feasibility Study (FS). All work will be performed in accordance with the United States Environmental Protection Agency (USEPA) -approved Field Sampling Plan (FSP), Quality Assurance Project Plan (QAPP), and Site-Specific Health and Safety Plan (HASP).

The PRP Group has prepared this Letter Work Plan based on discussions between the PRP Group and USEPA in February and April 2008. The Letter Work Plan incorporates comments received from USEPA on March 26, 2008 and May 5, 2008.

GROUNDWATER WORK OBJECTIVES

The general objectives for the phases of work discussed within this document include the following:

- define subsurface stratigraphy, including identifying till-rich zone(s) and sand and gravel aquifer zone(s) at locations beneath the Site to a depth of 100 feet below ground surface using Rotosonic drilling;
- collect data to assist in characterizing groundwater impact;
- recognizing that there may be seasonal or event-related differences in groundwater elevation, flow conditions and contaminant concentrations, and that there may be more

than one contaminant flow path and more than one source of groundwater contamination at the Site, attempt to: i) determine the appropriate screened interval(s) for shallow monitoring wells at Vertical Aquifer Sampling (VAS) locations through VAS data; ii) compare the screened intervals identified through VAS to the screened intervals and screen lengths in the existing wells; and iii) determine, based on these results and all existing data for the Site, if the screened intervals and screen length of the existing wells represent a zone of chemical impact in the shallow aquifer that is worthwhile to continue to monitor or not;

- characterize groundwater chemistry at Site monitoring wells through groundwater sampling and laboratory analysis; and
- collect groundwater and surface water elevation measurements over time to identify horizontal hydraulic gradients, flow directions, and, if nested wells are proposed in Phase 2, vertical hydraulic gradients.

Phase 1

In an effort to meet these objectives, Phase 1 will include three main work tasks VAS borings, synoptic water level measurements, and groundwater sampling for laboratory analysis.

1) VAS Borings

Figure 1 presents the locations of twenty-three on-Site VAS borings and two off-Site VAS borings (on the trailer park parcel). Additionally, the location of a soil boring that will be used to log the subsurface material below the large asphalt pile is presented on Figure 1. All of these borings, including the boring installed through the large asphalt pile, will be completed using Rotosonic drilling techniques. This drilling technique offers the opportunity to document relatively undisturbed soil sample cores, advance to the desired depth, and produces less waste than hollow stem auger drilling techniques. Additional details regarding Rotosonic drilling are provided in the FSP.

During borehole advancement, continuous soil cores will be observed, soil stratigraphy will be logged and cores will be screened with a photoioization detector (PID) for the presence of volatile organic compounds (VOCs), and screened for the presence of methane either by using a landfill gas meter (such as a Landtec GEM-500) or a flame-ionization detector (FID) calibrated for methane. Additionally, photographs will be taken of each 5-foot interval to obtain a photographic log of each borehole.

Core samples will be collected directly from the core barrel attached to the end of the drill string and extruded into cylindrical bags. Field measurements for VOCs and methane will be conducted along the cored material by piercing the plastic sleeve with the wand of the field instrument(s). In addition, the soils will be tested for the presence of non-aqueous phase liquids (NAPL) using the Sudan IV® dye test and/or another USEPA-approved shaker test, as appropriate. Field calibration, preventative maintenance, and SOPs for the PID and Sudan IV® dye test are included in the FSP.

Should the presence of NAPL be detected in a boring, the interval of detection will be recorded and advancement of the boring will be terminated to prevent introducing NAPL into deeper intervals. USEPA will be notified of the presence of NAPL at the location and the borehole location will be sealed in accordance with industry standards. Available stratigraphic information from such locations (up to and including the interval with detected NAPL) will be reviewed, and the location will be evaluated for additional work in Phase 2.

During borehole advancement, the amount of water added during Rotosonic drilling will be recorded. Every effort will be made to minimize the amount of water added during drilling in order to reduce the amount of purging required and to ensure that samples are representative of the groundwater in the aquifer formation. Groundwater samples will be collected at 5-foot intervals beginning at the 0 to 5-foot interval below the groundwater interface observed during borehole advancement. Groundwater samples will be collected from each discrete interval through a 5-foot long, stainless steel slotted screen using an inflatable packer with a submersible pump system. The flow rate for purging of groundwater will be dependent on the capacity of the submersible pump and the transmissivity of the aquifer material. Efforts will be made to maintain low flow during purging. Upon purging of two times the volume of water added during drilling (pre-purge), the flow rate will be reduced to the lowest sustainable flow rate and the minimum required screen volumes (i.e., three to five volumes of the 5-foot screened zone), will be purged. During the screen purging, field parameters such as pH, temperature, conductivity, oxidation-reduction (redox) reaction potential (ORP), dissolved oxygen (DO), and turbidity will be monitored to evaluate the stabilization of the purged groundwater. Groundwater samples will be collected once the parameters have stabilized as detailed in the FSP. VAS samples will not be collected from a 5-foot interval if attempts to purge and sample indicate the interval does not yield enough water to sample.

VAS will be completed to a depth of 100 feet below ground surface (bgs) at each location. All VAS samples will be analyzed for Target Compound List (TCL) VOCs,

total arsenic, and total lead. All of the groundwater samples collected during VAS and submitted to the laboratory will be unfiltered groundwater samples. In addition, VAS samples collected from select sampling intervals from each boring will be analyzed for TCL semi-volatile organic compounds (SVOCs) as discussed in further detail below. All of the groundwater samples collected during VAS and submitted to the laboratory will be unfiltered groundwater samples.

The sampling intervals that will be submitted for TCL SVOC analysis will depend on boring locations, whether the borehole is advanced through fill material (i.e., non-native material), or through native soil. The geophysical survey and, if the schedule permits, the test pit/test trench work that will be completed prior to the groundwater work discussed in this letter will help determine which VAS borehole locations are in fill material. The VAS borings determined to be located in fill material areas, or which have potential to be in fill material, will be completed first.

A total of four SVOC samples will be collected from each VAS boring as detailed below. In VAS borings drilled through fill (i.e., non-native) material, where the fill material extends below the water table, a maximum of three groundwater samples will be collected from the fill material for TCL SVOC analysis, and a minimum of one groundwater sample will be collected for TCL SVOC analysis from the native material directly beneath the fill material. The first sample in the fill material will be collected from the five-foot interval from the groundwater interface to five feet below the water table; subsequent groundwater samples collected from the fill material will be collected from every second five-foot interval. SVOC samples of native material will be collected at each five-foot interval commencing at the interface between the fill and native material. The total number of samples collected from the fill (i.e., non-native) material and from the native material at an individual VAS boring location will be dependant on the depth of fill material below the water table, i.e., if the fill material is sufficiently thick, three SVOC samples will be collected from the fill material and one from the native material, whereas if the fill material is thinner, fewer SVOC samples will be collected from the fill material and more samples will be collected from the native material (for a total of four SVOC samples per boring).

In VAS borings completed in native soil or where the fill material lies entirely above the water table, four samples for TCL SVOC analysis will be collected. The first sample will be collected from the five-foot interval beginning at the groundwater interface and the second from the interval from five feet below the water table to 10 feet below the water table. The third TCL SVOC sample will be collected at elevations corresponding to deeper areas of fill material below the water table. The fourth TCL SVOC sample will be

collected from the five-foot interval commencing at the elevation corresponding to the deepest fill material elevation observed in nearby borings advanced in non-native fill material. Sample elevations will be discussed with USEPA field representatives before starting VAS borings in areas believed to be in native soil areas.

The results of the VAS will be used to help select monitoring well locations (to be installed in Phase 2). The selection of monitoring well locations will be based on an analysis of VAS results and all existing data, including hydrostratigraphic data.

The proposed VAS borings are roughly laid out along four transects. The transects run approximately parallel to the section of the Great Miami River (GMR) northwest of the Site and continue toward the southeastern Site boundary. Following is a summary of the VAS boring locations, as identified along each transect, and the rationale for selecting each location. VAS boring locations may be revised based on the results of the Geophysical Survey and the Test Pit/Test Trench Investigation, which will be completed prior to the VAS sampling program if scheduling permits. Any modifications to the VAS boring and sampling program will be discussed with the USEPA prior to implementation.

Transect No.	*VAS Location No.*	*Rationale for VAS Location*
1	1	VAS location along northwest Site boundary to serve as a presumed upgradient data point. This location may be moved farther north along the transect, if possible, if fill is encountered.
	2	VAS location along northwest Site boundary and within 200 feet of MW-206 to evaluate aquifer data in vicinity of the well.

Transect No.	*VAS Location No.*	*Rationale for VAS Location*
1 cont'd.	3	VAS location along northwest Site boundary and within 200 feet of MW-201 and MW-103 to evaluate aquifer data in vicinity of these wells.
2	4	VAS location at northeast corner of Site boundary to serve as a presumed upgradient data point.
	5	VAS location to evaluate conditions in vicinity (or in presumed downgradient direction within vicinity) of former Dayton Recycling USTs. Off-set approximately 50 feet northwest of the transect.
	6	VAS location to evaluate conditions in vicinity (or in presumed downgradient direction within vicinity) of Valley Asphalt drum removal in 2000. Off-set approximately 100 feet northwest of the transect.
	7	VAS location to evaluate area presumed to be downgradient of material under the large asphalt stockpile. Off-set approximately 110 feet southeast of the transect.
	8	VAS location to evaluate area presumed to be downgradient of material under the large asphalt stockpile. VAS location to evaluate area downgradient of the large asphalt stockpile. Off-set approximately 275 feet southeast of the transect.
	9	VAS location to evaluate area presumed to be downgradient of material under the large asphalt stockpile. Off-set approximately 150 feet southeast of the transect.

2 cont'd	*VAS Location No.*	*Rationale for VAS Location*
	10	VAS location to evaluate the boundary between Parcel 5054 (Valley Asphalt) and Parcel 5177.
	11	VAS location to evaluate conditions at approximate center of PRPs' preliminary direct contact risk area (and located roughly 200-300 feet from former air curtain destructor).
	12	VAS location to evaluate presumed downgradient boundary of PRP Group's preliminary direct contact risk area.
	13	VAS location to collect data at southwest corner of Site boundary.
3	14	VAS location to evaluate conditions in vicinity of former Custom Delivery UST area. Off-set approximately 100 feet northwest of the transect.
	15	VAS location to evaluate aquifer conditions in vicinity of MW-202. Off--set approximately 225 feet southeast of the transect.
	16	VAS location to evaluate presumed downgradient boundary of PRP Group's preliminary direct contact risk area at northwest corner of Parcel 5176. Off-set approximately 225 feet southeast of the transect.
	17	VAS location to evaluate presumed downgradient boundary of PRP Group's preliminary direct contact risk area in vicinity of MW-203. Off-set approximately 100 feet southeast of the transect.
	18	VAS location to evaluate presumed downgradient boundary of PRP Group's preliminary direct contact risk area in vicinity of MW-101A and MW-204. Off-set approximately 200 feet northwest of the transect.
	19	VAS location within 200 feet of MW-209 and MW-212 to evaluate aquifer data in vicinity of these wells. If this location requires offsetting during field operations, it will remain at least 100 feet away from the edge of the Quarry Pond.
	20	VAS location to collect data south of the Quarry Pond.
4	21	VAS location to evaluate conditions within vicinity of MW-210. Off-set approximately 50 feet southeast of the transect.
	22	VAS location east of Quarry Pond to evaluate conditions at southeastern boundary of Site and Parcel 4423.
	23	VAS location to collect data at southeast corner of Site.

Two additional locations, 24 and 25, are proposed on the trailer park parcel to evaluate off-Site conditions in the presumed downgradient direction from MW-210.

The soil boring that will be used to log the subsurface material below the large asphalt pile will be advanced to a depth of 5 to 10 feet below the first native material encountered beneath the large asphalt pile (as determined in the field). The borehole will be advanced via Rotosonic drilling techniques, but VAS samples will not be collected from this borehole location. During borehole advancement below the large asphalt pile, continuous soil cores will be observed, soil stratigraphy will be logged and cores will be screened for the presence of VOCs and methane in the same manner as the VAS borings. A photographic log will also be compiled from each 5-foot soil core interval at this location.

Existing monitoring wells will be inspected, repaired as needed, and redeveloped to attempt to produce a silt free condition prior to water level monitoring and sampling. Redevelopment of wells and handling of investigative derived waste, including water from purging and pre-purging during VAS, will be performed in accordance with the USEPA-approved FSP.

2) Synoptic Water Level Measurements

Synoptic water level measurement events (groundwater and surface water) will be conducted in order to get a better understanding of groundwater flow directions. Note that staff gauges or measurement points will first be required for the GMR, Quarry Pond, and other surface water bodies. The reference elevations of the existing monitoring wells will be re-surveyed. Synoptic water level measurements will be completed using all permanent well installations and surface water measurement points once a month for the remainder of 2008. Any surface water measurement points that are disturbed during ongoing synoptic water level measurements will be immediately replaced and resurveyed. An oil/water interface probe will be used to monitor for the presence of light NAPL (LNAPL) in monitoring wells that are screened at the water table.

3) Groundwater Sampling

A round of groundwater sampling for TCL VOCs, TCL SVOCs, TCL pesticides and herbicides, TCL PCBs, and TAL metals will be completed at the existing monitoring wells. Groundwater sampling will be conducted using low flow field sampling procedures. The data will be compared with VAS results to assist in determining the adequacy of the existing monitoring wells.

The results from these three tasks will be summarized in a Technical Memorandum that, using new and existing data including representative hydrostratigraphic data and groundwater/surface water flow maps, will support and include the work proposed for Phase 2. The Technical Memorandum will be prepared following receipt of VAS analytical results and will contain only initial rounds of synoptic water level measurements. The Technical Memorandum will be reviewed in a project team workshop, similar to the meetings held with USEPA and Ohio EPA in early 2008.

Phase 2

Phase 2 will consist of three main work tasks – monitoring well installation, groundwater sampling, and continuous hydraulic monitoring.

1) Monitoring Well Installations

New monitoring wells will be installed based on the results of the Phase 1 VAS and all existing data, including hydrostratigraphic and groundwater/surface water flow data. If appropriate, the existing wells will be incorporated into the groundwater monitoring well network. All newly installed monitoring wells will be developed following installation. Following development, slug tests will be completed in each new monitoring well and in existing wells that will be kept/incorporated in the monitoring well network.

2) Groundwater Sampling

The Phase 2 groundwater sampling will include two rounds of sampling from the newly installed monitoring wells and, if appropriate, the existing wells. The first round of samples will be collected two weeks after installation and development of the monitoring wells and the second round will be collected two months later. The analyses will include TCL VOCs, TCL SVOCs, TCL pesticides and herbicides, TCL PCBs, and TAL metals, and monitored natural attenuation (MNA) parameters. The MNA parameters included in the analysis will be consistent with the USEPA Region 5 Monitored Natural Attenuation Framework. The complete list of MNA parameters is provided in Table K.3.3 of the QAPP. The analytical parameters may be reduced for the second round of sampling. The PRP Group will propose reductions in analytes, as appropriate, for USEPA's approval.

3) Continuous Hydraulic Monitoring

The monthly synoptic water level measurements described above would continue through Phase 2. More detailed hydraulic monitoring would be completed by installing transducers in select wells and surface water bodies. The transducers would provide continuous water level measurements that would aid in the evaluation of groundwater/surface water interactions. The data generated for this investigation would support the evaluation of remedial alternatives for the FS.

All work will be performed in accordance with the Field Sampling Plan, Quality Assurance Project Plan, and Site Specific Health and Safety Plan pending USEPA's approval of these documents.

SCHEDULE

Phase 1 fieldwork will be initiated within four weeks of USEPA approval of this Letter Work Plan, or the Field Sampling Plan, Quality Assurance Project Plan, and Site Specific Health and Safety Plan, and completion of the Geophysical Survey and, if the schedule permits, the Test Pit/Test Trench Investigation, whichever occurs later. The Phase 1 field tasks will be completed within a four-week period of time using two drill rigs working simultaneously. This schedule is subject to contractor availability and the actual drilling conditions encountered. The PRP Group will provide USEPA with written notification as much in advance as possible, but at least fifteen days in advance of the initiation of field activities. Phase 2 field work will begin following USEPA's approval of the Phase 1 Technical Memorandum. Monthly synoptic water level measurements will be taken throughout the remainder of 2008.

REPORTING

Phases 1 and 2 technical memoranda will be submitted to USEPA within two weeks of receipt of all data from the laboratory. The Phase 2 Technical Memorandum will provide a summary of results from monitoring well installation, groundwater sampling, and continuous hydraulic monitoring. Monthly progress reports during the Phase 1 and Phase 2 work will include the information required for monthly progress reports in the RI/FS SOW (including analytical data, groundwater/surface water elevations and stratigraphic information as it comes in).

Should you have any questions on the above, please do not hesitate to contact us.

Yours truly,

CONESTOGA-ROVERS & ASSOCIATES

Stephen M. Quigley

LA/ca/26
Encl.

c.c. Matt Mankowski, USEPA (PDF)
Matt Justice, Ohio EPA (PDF)
Eric Kroger, CH2M Hill (PDF)
Scott Blackhurst, Kelsey Hayes Company (PDF)
Wray Blattner, Thompson Hine (PDF)
Ken Brown, ITW (PDF)
Jim Campbell, Engineering Management Inc. (PDF)
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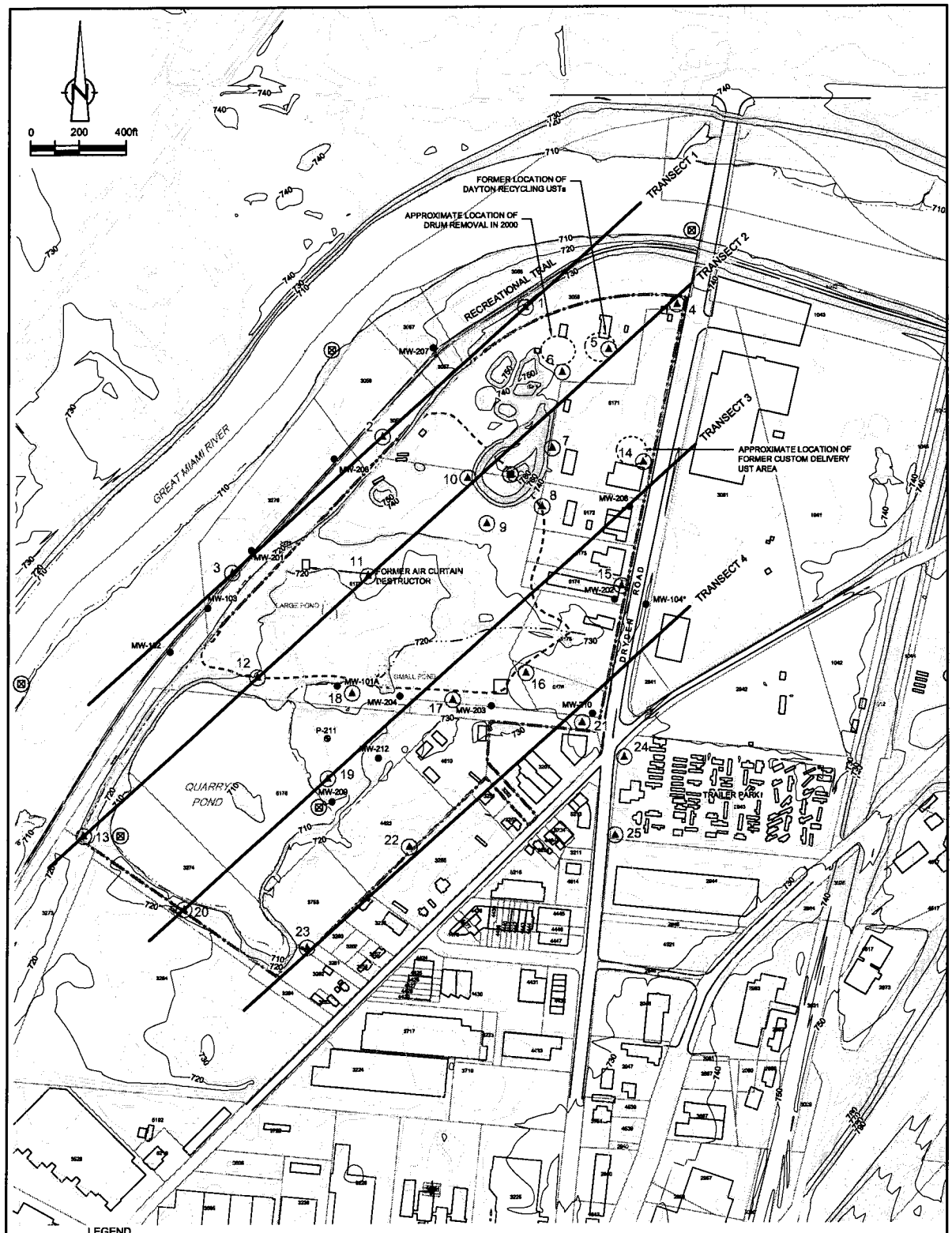
Paul Jack, Castle Bay (PDF)
Robin Lunn, Mayer Brown (PDF)
Roger McCready, NCR (PDF)
Karen Mignone, Pepe & Hazard (PDF)
Adam Loney, CRA (PDF)



LEGEND

- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- 730 EXISTING GROUND CONTOUR (2 FT CONTOUR INTERVAL)
- EDGE OF WATER
- MW-206 INTERMEDIATE ZONE MONITORING WELL LOCATION
- P-211 PIEZOMETER LOCATION
- PARCEL BOUNDARY
- 3264 LOT NUMBER
- APPROXIMATE LOCATION
- PROPOSED VAS SAMPLING LOCATION
- PROPOSED SURFACE WATER ELEVATION MONITORING LOCATION
- PROPOSED SOIL BORING LOCATION (TO LOG FILL MATERIAL)

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.

figure 1

VERTICAL AQUIFER SAMPLING LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-00(CIBU026)GN-WA001 MAR 28/2008

APPENDIX K-D

LANDFILL GAS/SOIL VAPOR INVESTIGATION LETTER WORK PLAN

CONESTOGA-ROVERS
& ASSOCIATES

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July 21, 2008

Reference No. 038443

Karen Cibulskis
Remedial Project Manager
United States Environmental Protection Agency
Region V
77 West Jackson Boulevard
Mail Code SR-6J
Chicago, IL 60604

Dear Karen:

Re: Final Landfill Gas/Soil Vapor Investigation Letter Work Plan
South Dayton Dump and Landfill Site, Moraine, Ohio (Site)

This Letter Work Plan presents the South Dayton Dump and Landfill Potentially Responsible Party Group's (PRP Group's) Work Plan for a landfill gas (LFG) and soil vapor investigation at the Site. A Site plan with proposed LFG/soil vapor sampling probe locations is provided on Figure 1. This work will help address data gaps and provide information to aid in the completion of a Feasibility Study (FS). All work will be performed in accordance with the United States Environmental Protection Agency (USEPA) -approved Field Sampling Plan (FSP), Quality Assurance Project Plan (QAPP), and Site-Specific Health and Safety Plan (HASP).

The PRP Group has prepared this Letter Work Plan based on the discussions between the PRP Group and USEPA in February 2008. The Letter Work Plan incorporates comments received from USEPA on May 7 and 28, 2008.

The objectives of this Letter Work Plan are to:

1. assess the presence of LFG and soil vapor at locations within the Site (pressure, methane, lower explosive limit (LEL), carbon dioxide and oxygen; and other chemicals at the detection limits listed in Table 1);
2. obtain current data in locations where historic information indicated potential landfill gas generation concerns;
3. develop information to assist in calculating future landfill gas generation rates for the FS. Four of the 20 gas probes are located within the limits of the Preliminary Direct Contact Risk - Presumptive Remedy Area (DC-PRA) and will provide information with respect to LFG/soil vapor generation within known municipal waste landfill areas at these locations. A total of 14 gas probe locations are proposed for installation along Dryden Road. Twelve of the 16 gas probes are located on commercial properties within 50 feet of occupied structures on Dryden Road. These gas probes will provide data near occupied structures; and

4. develop information to assist in evaluating the need for and type of landfill gas control at the Site for the FS.

LANDFILL GAS/SOIL VAPOR INVESTIGATION

Gas probes will be installed to evaluate LFG and soil vapor concentrations at locations within the Site, including the properties along Dryden Road. Twenty gas probes will be installed. Gas probe locations are presented on Figure 1. The procedures for installation of the gas probes are described below.

Five gas probes will be installed in the central portion of the Site (four within the DC-PRA) to evaluate the presence of methane and non-methane organic compounds (NMOC) in the zone where the LFG/soil vapors will most readily migrate at these locations. Three gas probes will be installed in the vicinity of the former underground storage tank removals and the Valley Asphalt drum removal area to assess landfill gas and soil vapor quality in the zone where the LFG/soil vapors will most readily migrate at these locations.

Fourteen of the gas probes are proposed to be installed on or adjacent to the Site boundary and in the vicinity of the commercial properties and structures along Dryden Road and west of East River Road to assess LFG and soil vapor quality in the zone where the LFG/soil vapors will most readily migrate and, if present, would pose the greatest risk to any occupants of the buildings at these locations.

GAS PROBE INSTALLATION

Gas probes will be installed using a 50-mm (2-inch) diameter Geoprobe dual-tube direct push technique to minimize formation disturbance. The borehole for each gas probe will be advanced to a target depth in the unsaturated zone [a maximum of 20 feet below ground surface (ft bgs) or 2 feet above the water table, whichever occurs first].

Soil and fill materials encountered will be logged. The soil log information recorded will include a visual description of the types of material (i.e., undisturbed native soil, spoils from quarry operations, domestic refuse, industrial refuse, metallic debris, ash, fly ash, construction and demolition debris, foundry sand, asphalt, slag, or other appropriate description) and, if possible, a Unified Soil Classification System (USCS) description. Native soils will be logged using the USCS by CRA's staff. A photograph of each core sample collected will be taken and a photographic log will be documented in the field notes. Should groundwater be encountered in any borehole, the tube will be pulled up a minimum of 2 feet above the water table. The void that is formed when the

tube is pulled will be filled using No. 3 silica sand. The groundwater elevation of the nearest monitoring well will be used to determine the targeted depth of the borehole for the gas probes.

LFG and soil vapor will not preferentially migrate through discrete intervals of fill material at the Site unless impermeable layers are present between the discrete intervals of fill material. Based on the available Site geological data, intervals that are impermeable to LFG/soil vapor have not been identified. Further, LFG and soil vapor migration to ambient air or into a building will occur from the shallow soil horizon. Accordingly, in areas where landfilled materials are not present, the screened interval of the gas probes will be installed in soil strata with a notably higher permeability than the surrounding geologic strata. The gas probe screen will be set as shallow as possible within the higher permeability stratum. In order to prevent short circuiting of ambient air into the gas probe and, consequently, dilution of LFG/soil vapor samples, the top of the gas probe screen will be installed a minimum of three feet below ground surface. The final depth of the gas probe screen will be dependent on the conditions observed at each location and will be determined in the field. The proposed soil vapor sampling program has been established to collect and analyze LFG/soil vapor samples that are representative of soil vapor quality in the most permeable zone in the vicinity of the probe, which is the zone where LFG and NMOC will migrate. If these soil borings encounter multiple, discrete permeable zones that appear to have vastly different LFG/soil vapor impacts based on field screening, then CRA will either consult with USEPA's field representatives and install more than one gas probe at that location or identify that area as potentially requiring additional characterization in later stages of investigation or remediation at the Site. The methods and procedures to be used for field screening will be provided in the FSP.

The average depth of the unsaturated zone across the Site is approximately 20 feet bgs; therefore, a target maximum depth of 20 feet bgs is based on the need to place the gas probes in the unsaturated zone near the surface where LFG/soil vapor, if present, will diffuse and migrate.

The purpose of this investigation is to assess the migration potential and generation rate(s) of methane and NMOC in the soil gas at sampled locations. If gas probes are installed in the 2-foot interval above the water table, the gas probes will periodically be saturated and will not generate meaningful data. The proposed gas probe locations will also address LFG/soil vapor concentrations at locations near potential receptors.

The screened interval will be selected based on field observations that will identify the presence of landfill materials or, in the absence of such materials, a comparatively permeable region in the unsaturated zone that would be expected to transmit LFG and/or soil vapor. The selection of the most permeable zone will be based on soil descriptions and characterizations using the Unified Soil Classification System (USCS). The gas probe sampling and screened interval selection details are summarized in the Field Sampling Plan (FSP), CRA May 2008. Where landfilled materials are present, the screen will be placed at a depth immediately above the landfilled materials. If the landfilled material extends to within three feet of the surface and it is, therefore, not possible to set the screen above the landfilled material, the screen will be placed within the landfilled material, with

the screened interval set as close to the top of the landfilled materials as possible but deep enough to minimize the breakthrough of ambient air from the surface (i.e., 3 to 5 feet bgs).

The gas probes will be completed using 13-mm (0.5-inch) diameter schedule 40 PVC continuous piping (i.e., no joints) with a screened interval length of 0.3 meters (1 foot). The void space between the screened interval and formation will be filled with No. 3 silica sand (i.e., sand pack) to approximately 0.2 meters (8 inches) above the top of the screened interval. One foot of dry granular bentonite will be placed on top of the sand pack and then hydrated bentonite will be placed to just below ground surface. The sand pack and bentonite seal will be placed as the Geoprobe is withdrawn to ensure that the formation does not collapse around the screened interval or riser. A lockable surface casing will be set in concrete at the ground surface around each gas probe. The gas probe completion details are summarized in the FSP. The gas probe stratigraphic and instrumentation logs are presented in the FSP.

Soil samples will be collected from the surface and subsurface during the gas probe installation for the analysis of soil physical properties (i.e., grain size analyses, fraction of organic carbon content, plasticity index, porosity, permeability, and Atterburg limits). The procedures for collecting soil samples are presented in the FSP.

LANDFILL GAS/SOIL VAPOR SAMPLING

CRA will complete two rounds of sampling. The sampling will consist of:

i) measurement of gas pressure;

ii) screening for methane (v/v), LEL, and oxygen (v/v); and

iii) collection of Summa™ canister samples for VOC analysis.

The initial LFG/soil vapor sampling will be conducted one week following the installation of gas probes. One week is considered to be more than sufficient time for any formation disturbances created by drilling activities to dissipate and for equilibrium conditions to be reestablished in the unsaturated zone. As a result, the soil vapor samples are considered representative of conditions in the sampled intervals at the time the samples are collected.

Soil gas sampling will not be performed during or within 48 hours of a significant rainfall event [e.g., >0.5 inches after California Environmental Protection Agency (CalEPA, 2003)]. This will help avoid the potential that increased moisture content in the unsaturated zone soil could temporarily dampen soil gas concentrations, or possibly prevent soil gas sample collection (i.e., such as in cases where the soil gas probe screened interval could become temporarily saturated due to the passing infiltration front). In fine-grained soil conditions, consideration will be given to allowing a greater

amount of time for rainfall events to dissipate. The potential influence of rainfall events on soil gas concentrations is less of a concern in cases where the soil gas probes are located beneath impervious ground cover (e.g., pavement or building foundation).

The three sampling elements are described below.

i) Measurement of Gas Pressure

A pressure gauge will be attached to the hose barb on the LFG probe to measure the static gas pressure. The pressure gauge will be sufficiently sensitive to record gas pressure to 0.1 pounds per square inch (psig). The highest value obtained during gas pressure readings will be recorded. The ambient barometric pressure will be recorded at each gas probe when soil gas pressure readings are being taken. The ambient barometric trends will also be noted (i.e., rising, falling, steady).

Two rounds of gas pressure measurements will be collected, separated by at least one month.

ii) Screen for Methane, LEL, Carbon Dioxide, and Oxygen

A Multimeter will be used to draw a sample from each probe to measure and record the methane, LEL, carbon dioxide, and oxygen readings. The highest values obtained during sampling will be recorded. The ambient and soil gas temperatures will be recorded at each gas probe when soil gas readings are being taken. The ambient barometric trends also will be noted (i.e., rising, falling, or steady).

Two rounds of this sampling will be completed, separated by at least one month.

The details regarding the calibration and maintenance frequency and procedures, instrument start up procedures, and recording of data for instruments used during the installation and sampling of the gas probes will be provided in the FSP. These instruments include PIDs, Multimeters, barometers, and thermometers. The FSP will specify gas probe purging rates and procedures. A copy of the supplier instrument calibration will be available for review in the field. All field calibration procedures and readings will be documented in the field logbook.

iii) Summa™ Canisters

One round of soil vapor samples will be collected during the first round of methane measurements using 6-liter capacity Summa™ canisters fitted with a laboratory calibrated critical orifice flow regulation device sized to allow the collection of the soil vapor sample over a 1-hour sample collection time. The Summa™ canisters will be fitted with a laboratory calibrated critical orifice flow regulation device sized to restrict the maximum soil gas sample collection flow rate to approximately 100 milliliters per minute (mL/min), which corresponds to the lower end of the maximum soil gas sampling flow rate of 100 to 200 mL/min recommended by CalEPA (CalEPA,

2003). A flow rate of 100 mL/min is recommended to limit VOC stripping from soil, and prevent the short-circuiting of ambient air from ground surface that would dilute the soil vapor sample. The low flow rate of 100 mL/min will increase the likelihood that a sample representative of in situ conditions is obtained. Prior to sample collection, gas probe purging will be conducted at a maximum flow rate of 200 mL/min. Three gas probe volumes (calculated based on casing and sand pack volume) will be purged to remove potentially stagnant air from the internal volume of the gas probe. The FSP provides the soil gas purging and sampling procedures including the calculation of purge volume, maximum purge volume and maximum purging rates. Once the flow rate is set for a canister, the time it will take to fill up the canister will be calculated and the sampler will retrieve the canister and turn off the flow at the calculated time to prevent the valve from being open after the canister is filled.

The Summa™ canister samples will be analyzed for VOCs using USEPA method TO-15. The VOCs included in USEPA method TO-15 (with the addition of naphthalene) and the best method detection limits that the contract laboratory can achieve are listed in Table 1. The laboratory's ability to achieve the best possible detection limits will be highly dependent on the presence of matrix interferences.

Quality assurance /quality control (QA/QC) measures to be implemented during the soil vapor sampling event include maintaining a minimum negative pressure in the Summa™ canisters following sample collection, collection of one field duplicate sample, collection of an ambient air sample, and the analysis of a trip blank Summa™ canister. Further details regarding the gas probe sampling protocol and the applied QA/QC measures are presented in the FSP.

SCHEDULE

The LFG and soil vapor investigation will begin within four weeks of USEPA approval of this Letter Work Plan, or the relevant sections of the Field Sampling Plan and Quality Assurance Project Plan, or USEPA's review of the Health and Safety Plan, whichever occurs later and following completion of clearing and grubbing activities and, if scheduling permits, test pitting and test trenching activities. The LFG and soil vapor investigation will be completed over a two-week period. The second LFG sampling event (gas pressure, methane, LEL, and oxygen) will occur within six weeks of the first sampling event. The PRP Group will provide the USEPA with verbal notification at least 15 days in advance of the initiation of this activity.

All work will be performed in accordance with the FSP, QAPP, and HASP, pending USEPA's approval of the relevant sections of these documents.

REPORTING

The results of the LFG and soil vapor investigation and analytical results will be summarized and presented in a technical memorandum. The memorandum will include a description of the fieldwork completed, any deviations from the proposed work, and the rationale behind the change, and photographs taken during the investigation. Figures detailing the actual installations, analytical summary tables, iso-concentration maps, and analytical data reports will also be included in the technical memorandum. The technical memorandum will be provided to the USEPA within one month of the completion of the proposed work. The data will be used in the FS and to assist in identifying potential areas where further investigation or assessment may be appropriate.

Should you have any questions on the above, please do not hesitate to contact us.

Yours truly,

CONESTOGA-ROVERS & ASSOCIATES

Stephen M. Quigley

AL/ca/30
Encl.

c.c. Matt Mankowski, USEPA (PDF)
Matt Justice, Ohio EPA (PDF)
Brett Fishwild, CH2M Hill (PDF)
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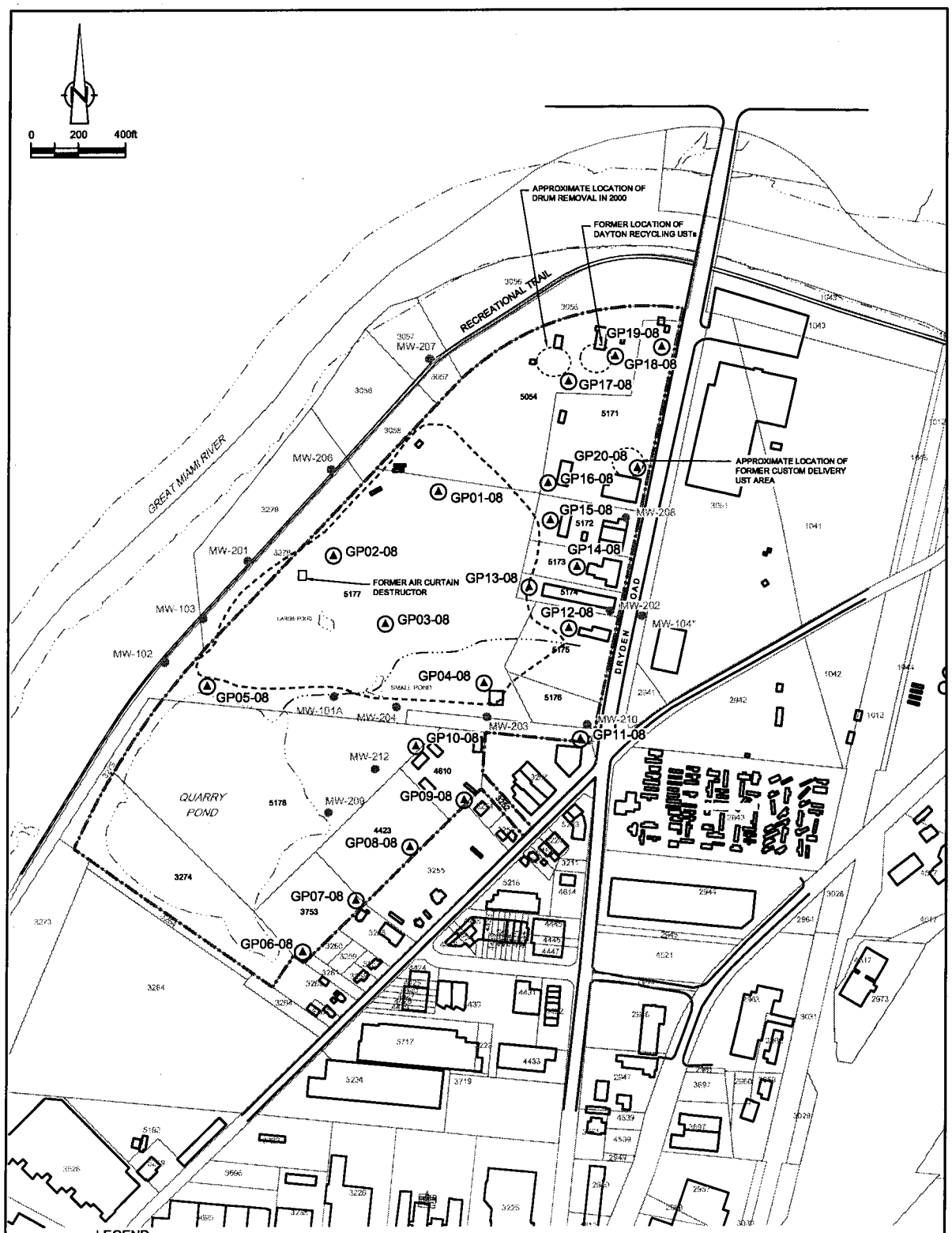

LEGEND

- MW-206 INTERMEDIATE ZONE MONITORING WELL LOCATION
- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- EDGE OF WATER
- GP01-08 PROPOSED LANDFILL GAS PROBE LOCATION
- APPROXIMATE LOCATION

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
USGS AERIAL PHOTOGRAPH, DAYTON SOUTH, 1994.

figure 1

SOIL GAS PROBE LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-60(CIBU041)GN-WA001 JUL 14/2008

TABLE 1

SOIL GAS PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1] *Air* ($\mu g/M^3$)	*Method Detection Limits (MDL)* [2] *Air* ($\mu g/M^3$)	*OSWER Draft Guidance* *Targeted Soil Gas Concentrations* [3] *Risk = 1 × 10⁻⁴* ($\mu g/M^3$)
Select Volatile Organic Compounds (VOC)			
Acetone	24	5.9	3,500
Benzene	0.96	0.64	310
Bromodichloromethane	2.0	1.6	140
Bromoform	4.1	2.1	2,200
Bromomethane	16	7.8	50
2-Butanone	29	5.9	10,000
Carbon disulfide	31	6.2	7,000
Carbon tetrachloride	1.9	1.3	160
Chlorobenzene	1.4	0.92	600
Chloroethane	1.1	1.0	100,000
Chloroform	1.5	0.97	110
Chloromethane	1.6	0.82	900
Cyclohexane	1.7	1.4	N/A
Dibromochloromethane	3.4	1	100
1,2-Dibromo-3-chloropropane	9.6	3.9	2
1,2-Dibromoethane	3.1	1.5	2
1,2-Dichlorobenzene	2.4	1.2	2,000
1,3-Dichlorobenzene	2.4	1.2	1,100
1,4-Dichlorobenzene	2.4	1.2	8,000
Dichlorodifluoromethane	1.5	0.99	2,000
1,1-Dichloroethane	1.2	0.81	5,000
1,2-Dichloroethane	8.1	4.0	94
1,1-Dichloroethene	7.9	4.0	2,000
cis-1,2-Dichloroethene	7.9	3.2	350
trans-1,2-Dichloroethene	7.9	4.0	N/A
1,2-Dichloropropane	14	6.9	40
cis-1,3-Dichloropropene	1.8	0.91	200
trans-1,3-Dichloropropene	1.8	0.91	200
Ethylbenzene	1.3	0.87	2,200
2-Hexanone	2.0	1.6	N/A
Isopropylbenzene	2.5	2.0	4,000
Methylene chloride	1	0.69	5,200
4-Methyl-2-pentanone	41	8.2	800
Methyl tert-butyl ether	7.2	3.6	30,000
Naphthalene	2.6	1.3	30
Styrene	1.7	0.85	10,000
1,1,2,2-Tetrachloroethane	14	6.8	42
Tetrachloroethene	2.7	1.4	810
Toluene	7.5	3.8	4,000
1,2,4-Trichlorobenzene	37	18	2,000
1,1,1-Trichloroethane	1.6	1.1	22,000

TABLE 1

SOIL GAS PARAMETER LISTS AND TARGETED QUANTITATION LIMITS
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
MORAINE, OHIO

Parameter	*Targeted Quantitation Limit (TQL)* [1] *Air* ($\mu g/M^3$)	*Method Detection Limits (MDL)* [2] *Air* ($\mu g/M^3$)	*OSWER Draft Guidance Targeted Soil Gas Concentrations* [3] *Risk = 1 × 10⁻⁴* ($\mu g/M^3$)
Select VOC (continued)			
1,1,2-Trichloroethane	1.6	1.1	150
Trichloroethene	2.1	1.1	22
Trichlorofluoromethane	11	5.6	7,000
1,1,2-Trichloro-1,2,2-trifluoroethane	3.8	3.1	300,000
Vinyl chloride	7.6	3.8	280
Xylenes (total)	17	4.3	70,000

Notes:

1 Please note that these are targeted quantitation limits and are presented for guidance only. Actual quantitation limits are highly matrix dependent and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes.

2 Method Detection Limits (MDL) are also presented for guidance only. Actual MDLs will vary depending on sample specific preparation factors. The MDLs are also highly matrix dependant and may be elevated due to matrix effects, QA/QC problems and high concentrations of target and non-target analytes. Laboratory MDLs are updated on a periodic basis and the MDLs in effect when the samples are analyzed will be used for reporting purposes.

3 Target Shallow Soil Gas Concentrations Corresponding to Target Indoor Air Concentrations Where the Soil Gas to Indoor Air Attenuation Factor = 0.1 in Table 2a (Risk = 1×10^{-4}) of draft guidance "Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils" (USEPA, 2002).

APPENDIX K-E

LEACHATE SEEP INVESTIGATION LETTER WORK PLAN

CONESTOGA-ROVERS
& ASSOCIATES

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May 6, 2008

Reference No. 038443

Ms. Karen Cibulskis
Remedial Project Manager
United States Environmental Protection Agency
Region V
77 West Jackson Boulevard
Mail Code SR-6J
Chicago, IL 60604

Dear Karen:

Re: Final Leachate Seep Investigation Letter Work Plan
South Dayton Dump and Landfill Site, Moraine, Ohio (Site)

This Letter Work Plan presents the South Dayton Dump and Landfill Potentially Responsible Party Group's (PRP Group's) Work Plan for a Leachate Seep Investigation at the Site. A Site plan showing Site topography, including embankments, is provided on Figure 1. This work will help address data gaps and provide information to aid in the completion of a Feasibility Study (FS).

The PRP Group has prepared this Letter Work Plan based on the discussions between the PRP Group and USEPA in February and April 2008. The Letter Work Plan incorporates comments received from USEPA on April 2 and May 1, 2008.

The objectives of this Work Plan are to:

1. complete a seep inspection to identify the location, extent, and characteristics of seeps observed along Site embankments and in other on-Site and near-Site areas;
2. characterize seeps observed along Site embankments and in other areas; and
3. identify any area(s) that may require further investigation.

The work associated with achieving these objectives is described further below.

VISUAL SEEP INSPECTION

CRA will complete a visual inspection of:

- the embankments and nearby areas on the west side of the Site (adjacent to the Great Miami River);

- embankments and nearby areas to the north including to the north of the Valley Asphalt property;
- areas surrounding the Quarry Pond;
- embankments and nearby areas along the central access road;
- embankments and nearby areas in the vicinity of the air curtain destructor;
- embankments and the area in the vicinity of the Small Pond; and
- embankments and the area in the vicinity of the Large Pond.

This assessment will consist of a visual inspection of the entire embankment surface, nearby areas, and low lying areas with an objective to document any evidence of groundwater or leachate discharge from any portion of the bank and other nearby or low-lying areas. Specific items to be investigated include identifying erosion rills, areas of surface staining and/or stressed vegetation, and wet or saturated areas resulting from seeping liquid.

CRA will prepare a photographic log for the inspection. The photographic log will list the date of each photograph, a specific description of what the photograph depicts, its location, and the photographer.

Seep inspections will not be performed during precipitation events and will be performed no sooner than 24 hours after a precipitation event. To the extent practicable, given the project schedule and USEPA notification requirements, the PRP Group will schedule the seep inspection to occur after several days of dry conditions (based on long term weather forecasts). In the event of precipitation during the seep inspection, field activities will be suspended and will not recommence until 24 hours after the rain has ceased. The USEPA will be notified of any delays in the seep inspection. Also the weather conditions will be noted in the daily field logs.

Potential seeps encountered during the Survey, Geophysical Investigation, or other Site work will be flagged, and these areas will be inspected during the seep inspection if the potential seep is encountered prior to the Leachate Seep Investigation or at a later date if the potential seep is found after the Leachate Seep Investigation and does not correspond to a previously identified seep.

SEEP CHARACTERIZATION

Should leachate seeps, surface staining, stressed vegetation, or other evidence of a leachate seep be identified in any of the embankments or in other areas, CRA will flag the location and survey it using a hand-held global positioning system (GPS) device and record the coordinates. CRA will then record the characteristics of each seep area including color of staining; area of staining; whether

the seep is active or not active; estimate of seep flow; color of seep flow; presence of erosion, pooling, or odor; PID reading; and any other pertinent or identifying details. CRA will also record potential downgradient receptors for each seep, such as landfill interior (where capping alternatives will be evaluated in the FS), the Great Miami River, Quarry Pond, etc. After surveying the location and recording seep observations, CRA will immediately proceed to collect leachate and/or soil samples (as detailed below) at the identified location before continuing on to the next area.

If an active seep is observed, liquid sampling will be attempted. The area located immediately beneath the seep will be dug out using a clean shovel or trowel. A clean sample jar or pail will be set into the dug out area and the liquid will be allowed to accumulate in the container. The liquid will be transferred to sample containers for submission to the analytical laboratory. As the volume of liquid may be limited, prioritization of requested analyses for the sample will be as follows: Target Compound List (TCL) volatile organic compounds (VOCs), Target Analyte List (TAL) metals and cyanide, TCL semi-volatile organic compounds (SVOCs), TCL pesticides, and TCL polychlorinated biphenyls (PCBs).

CRA will attempt to place the sample jar or pail on an angle in order to encourage leachate to flow into the jar rather than dripping in. VOC sample vials will be filled by slowly, smoothly, and carefully transferring seep water from the large clean sample jar to the VOC vial, without splashing, and sealing the vial to ensure that no air bubbles are allowed to remain in the vial. VOC sampling will be conducted first, once sufficient liquid has been allowed to collect in the large clean sample jar. Trip blanks, field blanks and duplicate samples (if sufficient sample is available) will be collected in conjunction with the seep sampling. Trip blanks will be submitted with each sample shipment to the analytical laboratory. Field blanks will be collected at a frequency of one per every ten seep samples collected. Field duplicates will be collected at a frequency of one per twenty seep samples (sample volume permitting). The Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP) provide additional details and instruction on sample collection, preservation, and quality assurance/quality control.

If a sufficient volume of liquid to fill sample jars is not produced by the seep, CRA will collect a sample of the surface soil in the area of the seep. The soil sample will be collected from a saturated portion of the soil immediately beneath the seepage. The surface soil sample will be collected as part of the leachate seep investigation fieldwork and will be analyzed for TCL VOCs, TCL SVOCs, TCL pesticides, TCL PCBs, TAL metals, and asbestos.

If no active seep is observed but indirect evidence of a seep is observed (erosion rills, stressed vegetation, etc.), then CRA will collect a surface soil sample from the area where the observation was made. The soil sample will be analyzed for TCL VOCs, TCL SVOCs, TCL pesticides, TCL PCBs, TAL metals, and asbestos.

All work will be performed in accordance with the FSP, QAPP, and Site-Specific Health and Safety Plan (HASP) pending USEPA's approval of these documents.

IDENTIFY AREAS NEEDING FURTHER INVESTIGATION

The field observations and analytical data generated from any liquid seep or soil sampling will be reviewed and evaluated. Areas where stressed vegetation was observed may be considered as alternative sampling areas for the Test Pit/Test Trench Investigation. Analytical data will be evaluated against USEPA Region 9 Preliminary Remediation Goals (PRGs). If liquid or soil analytical data indicate that there are constituents present at concentrations greater than Region 9 PRGs, then the area where the sample was collected may require further investigation or assessment for the FS. If liquid or soil sample data do not exceed Region 9 PRGs, then the area where the sample was collected will not require further leachate seep assessment for the purpose of completing the FS. Additional leachate seep assessment at these locations may, however, be required as part of Remedial Design (e.g., to evaluate seasonal and/or yearly fluctuations in leachate seeps).

If the soil contains constituents at concentrations greater than the applicable Ecological Screening Criteria, and the seep area is outside the area to be evaluated for capping alternatives, then the area may require further assessment as part of the RI/FS for areas not addressed by the FS. If the seep is in the interior of the landfill (where capping alternatives will be evaluated in the FS), then the area will be noted and evaluated as part of the FS. The assessment and evaluation of data generated as part of this investigation will be presented in a technical memorandum. Modification or adjustments to further investigative work proposed for the Site in 2008 will be discussed with the USEPA prior to implementation.

SCHEDULE

The leachate seep inspection will begin within two weeks of USEPA approval of this Letter Work Plan, or the relevant sections of the FSP and QAPP, or USEPA's review of the HASP, whichever occurs later, and will be completed over a two-day period of time (weather permitting). The PRP Group will provide the USEPA with verbal notification 15 days in advance of the initiation of this activity, and will use extended weather reports in an attempt to time the event during dry weather or no sooner than 24 hours after a precipitation event.

REPORTING

The results of the seep inspection and any analytical results (if samples are collected) will be summarized and presented in a technical memorandum. The memorandum, which will include a

description of the field work completed, any deviations from the proposed work and the rationale behind the change, photographs, a figure identifying areas inspected, a figure showing the location of identified seeps indicating which seeps, if any, were active at the time of the inspection, analytical summary tables, and analytical data reports, will be provided to the USEPA within one month of the completion of the proposed work. The technical memorandum will also include a table including seep descriptions and approximate elevations (from the Site survey). The data will be used in the FS and to identify potential areas where further investigation or assessment may be appropriate.

Should you have any questions on the above, please do not hesitate to contact us.

Yours truly,

CONESTOGA-ROVERS & ASSOCIATES

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AL/ca/27
Encl.

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Tim Hoffman, Representing Kathryn Boesch and Margaret Grillot (PDF)
Paul Jack, Castle Bay (PDF)
Robin Lunn, Mayer Brown (PDF)
Roger McCready, NCR (PDF)
Karen Mignone, Pepe & Hazard (PDF)
Adam Loney, CRA (PDF)

LEGEND

- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- EXISTING GROUND CONTOUR (2 FT CONTOUR INTERVAL)
- EDGE OF WATER
- PARCEL BOUNDARY
- AREA OF LEACHATE SEEP SURVEY

figure 1

LEACHATE SEEP INVESTIGATION
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.

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FIELD AND LABORATORY STANDARD OPERATING PROCEDURES

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iii)	Turbidity	38443--NTU-02
iv)	Dissolved Oxygen	38443--DO-02
v)	Oxidation Reduction Potential	38443--ORP-02
vi)	Soil VOC Screening	FP-VOC-02
vii)	Field Procedures for Measuring Iron II(Ferrous)	FP-IronII-01
II.	**LABORATORY SOPs**	
A.	Support Procedures – TestAmerica North Canton	
i)	Sample Receiving and Sample Control	NC-SC-0005
ii)	Glassware Washing	NC-QA-0014
iii)	Nonconformance and Corrective Action System	CORP-QA-0010
iv)	Quality Control Program	QA-003
v)	Records Information Management	NC-QA-0019
vi)	Statistical Evaluation of Data and Development of Control Charts	NC-QA-0018
B.	Support Procedures – TestAmerica West Sacramento	
i)	Sample Receipt and Procedures	WS-QA-0003
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iv)	Quality Control Program	WS-PQA-003
v)	Document Archiving	WS-QA-0009

C. Support Procedures – TestAmerica Los Angeles

i)	Sample Receiving, Login, and Internal Chain of Custody for Air Samples	LA-SRA-001
ii)	Glassware Washing	NA
iii)	Nonconformance and Corrective Action System	LA-QAS-006
iv)	TestAmerica Los Angeles Quality Control Program	LA-QAP-002
v)	Corporate Document Control and Archiving	CW-Q-S-001
vi)	Statistical Evaluation of Quality Control Data and Development of Control Charts	LA-QAS-001

D. Support Procedures – EM Labs

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	ii)	Extraction of Organic Compounds From Water and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series Methods	NC-OP-032
	iii)	Extraction Procedure for Chlorinated Acid Herbicides Based on Method 8151A	NC-OP0031
	iv)	GC/MS Analysis Based on Methods 8270C	CORP-MS-0001NC
	v)	GC Analysis Based on Methods 8000B, 8021B, 8081A, 8082, 8151A, 8141A, 8015B, and 615	NC-GC-038
	vi)	Analysis of Dissolved Gases in Groundwater by Modified Method RSK-175	NC-GC-0032
	vii)	Method 8290 and TO-9A Polychlorinated Dioxins and Furans by HRAC/HRMS [Method 8280]	WS-ID-0005
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iv)	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-8467470A and MCAWW 245.1.	CORP-MT-0005NC
v)	Acid Digestion of Soils, SW846 Method 3050B	CORP-IP-0002NC
vi)	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, Method SW-846 7471A	NC-MT-011
G.	Inorganics Preparation and Analysis	
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ii)	pH Electrometric Method	NC-WC-0010
iii)	Total Organic Carbon (TOC)	NC-WC-0017
iv)	Cyanide Preparation Method	NC-WC-0032
v)	Cyanide Automated, Pyridine-Barbituric Acid	NC-WC-0031
vi)	Sulfide	NC-WC-0060
vii)	Flashpoint Closed Cup	NC-WC-0034
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ix)	Determination of Inorganic Anions by Ion Chromatography	NC-WC-0084
H.	Soil Gas Preparation and Analysis	
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III.	**CRA Data Validation SOP**	
	i) Analytical Data Quality Assessment and Validation Standard Operating Procedures (SOP)	Jan. 2008, Rev. 2

pH/TEMPERATURE

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, revised March 1983, Method 150.1

Sensitivity: 0.01 pH unit; 0.1°C

Optimum Range: pH 1.00 to 12.00; temperature -5 to 50°C

Sample Handling: Determined on site

Reagents and Apparatus:

1. Temperature compensated pH meter, Water Quality Monitoring System;
2. Combination pH electrode;
3. Thermilinear thermister temperature probe;
4. pH buffer solutions, pH 4.00, 7.00, and 10.00 (certified buffer solutions);
5. Distilled or deionized water in wash bottle.

Calibration:

1. Switch On/Off key to on. Before connecting the pH electrode, zero the electronics with the shorting cap attached to the meter. Turn on the meter and set the pH function switch to pH. Connect the shorting cap to the pH-input jack and set the manual temperature compensation knob to 25°C. Adjust the CAL control to indicate 7.00 ±0.01 on the pH-mV display. Disconnect the shorting cap from the pH input and connect it to the mV-input jack. The monitor is now zeroed.

2. Test the pH electrode for noise and offset as follows: Rinse the Temperature Probe with pH 7.00 buffer to remove any contaminants. Connect to the pH-input jack and to the TEMP input jack. Pour pH 7.00 buffer into a 50 mL sample cup then immerse both of the sensors into the buffer at 25.0 ±0.1°C (use the °C display to confirm the temperature). Allow the sensors to equilibrate. A display value other than 7.00 shows electrode background noise and offset. The background noise and offset at pH 7.00 should not exceed ±0.2 pH units at 25°C. Replace pH probe if background noise exceeds this tolerance.

3. Set the function switch to pH ATC. Connect to the pH ATC input jack. The pH ATC function will not work unless it is connected to the pH ATC input.

4. Rinse the Temperature Probe with pH 7.00 buffer to remove any contaminants. Connect to the pH-input jack and to the TEMP input jack. Pour pH 7.00 buffer into a 50 ml sample cup; immerse both of the sensors into the buffer. Allow the sensors to equilibrate in the buffer until a stable reading is obtained. Read the temperature and adjust the pH manual temperature compensation knob to the same value. Adjust the CAL control knob for 700 ±0.01 pH units in the display and discard the buffer. Rinse the sensors with deionized or distilled water, followed by a rinse of the next desired buffer (typically pH 4.00 or 10.00). Half fill another disposable 50 ml sample cup with the next buffer for calibration and immerse the sensors. Allow the sensors to equilibrate until a stable reading is obtained. The temperature of the two buffers should not differ by more than ±0.1°C. Adjust the SLOPE control until the display is within 0.01 pH units of the buffers, stated value. Discard the buffers. The pH system is now calibrated and ready for use.

Procedure:

1. Calibrate meter using calibration procedure.

2. Set up meter as outlined in the operating manual.

3. Pour the sample into clean sample jar or plastic cup.

4. Record temperature and pH of the sample in the logbook.

5. Rinse with water and pH 7.00 buffer.

6. Repeat steps 3 through 5 for each sample.

7. Recheck calibration with pH 7.00 buffer solution after every 10 or fewer samples and after the last sample.

8. Store pH electrode in soaker bottle when not in use.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be ±0.2 pH units.

   If the results are outside of the control limits, rinse electrodes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If drift is suspected to be the cause of

the problem, clean the electrode and recalibrate. If drift is still apparent, replace electrode.

2. Calibration check results must be ±0.10 pH unit of the true value. If the result is outside of ±0.10 pH unit, rinse electrodes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in-control calibration.

3. All glassware is to be soap and water washed, tap water rinsed and distilled or deionized water rinsed prior to analyses.

Interferences:

Interferences in pH measurements occur with presence of weak organic and inorganic salts and oil and grease. If oil and grease are visible, note in logbook. Clean electrode with soap and water, followed by 10% HCl and deionized water rinse. Recalibrate meter before analysis of next sample.

SPECIFIC CONDUCTANCE

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance

Reference: "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, revised March 1983, Method 120.1

Sensitivity: 0.1 mmhos/cm

Optimum Range: 0 - 100.0 mmhos/cm

Sample Handling; Determine on site

Reagents and Apparatus:

1. Conductivity meter - YSI Model Water Quality Monitoring System;
2. Conductivity Cell - YSI Model Flow-Through Conductivity Cell (K=5/cm);
3. Thermilinear Thermister - YSI Model Temperature Probe;
4. Deionized water;
5. Conductivity standard, 1.0mmho/cm @25°C.

Notes:

The conductivity meter is factory calibrated. The calibration is checked using a solution of known conductance.

Calibration Check

Connect the cell and the Temperature Probe, and remove them from the sample chamber. Set the conductivity function switch to 2 ATC. Rinse the inside and outside of the cell and the probe with about 1/3 the calibration solution. Place both of the sensors into the remainder of the solution in the bottle and allow them to come to temperature equilibrium. Make sure that the body is immersed so that the liquid level is half way up the knurled portion of the cell. Read the displayed value and determine if the cell/instrument is within specified accuracy. The displayed value is corrected to 25°C automatically and should be 1.000 ±070 mmho/cm. If the value is not within specification replace cell.

Procedure:

1. Check calibration of meter.

2. Set up meter as outlined in the operating manual.

3. Before any conductivity cell is used, it should be soaked in distilled or deionized water for at least one hour. To make conductivity measurements, connect a YSI Flow-Through Conductivity Cell to the monitoring system. Set the conductivity function switch to 2 and observe the displayed value after the reading is stable. The display reads out in mmho/cm.

4. If the overrange signal (1.9999) is displayed, the conductivity of the water being measured is greater than 1.999 mmho/cm. Reset the function switch to 20. If the overrange signal is still displayed, reset to 100. If the overrange signal is still displayed, either the conductivity is greater than 100.0 mmho/cm and the YSI Water Quality Monitor can not be used for conductivity determinations.

5. Record conductance readings in field logbook.

6. Repeat steps 3 through 5 for remaining samples.

Quality Control:

1. The quality control calibration check standard must be analyzed initially, after every 10 or fewer samples and after the last sample. If less than 10 samples are analyzed, the calibration standard is still required to be analyzed. The standard must be within ±10 percent of the true value or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standard in the field logbook.

2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicate values are to be within ±15% of each other. If outside of this range, reanalyze the samples. If still outside the acceptance range, recollect sample and reanalyze. If still out, replace probe.

TURBIDITY

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Nephalometric

Reference: "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, revised March 1983, Method 180.1

Sensitivity: 0.01 Nephalometric Turbidity Unit (NTU)

Optimum Range: 0 - 10; 0 – 100; 0-1,000 NTU

Sample Handling: Determined on site

Reagents and Apparatus:

1. Direct reading turbidity meter, HF Scientific Model DRT-15CE, HACH 2100P, and LaMotte;
2. Cuvettes with screw tops;
3. Battery charger;
4. 0.02 NTU (nominal) reference standard;
5. Distilled or deionized water in wash bottle.

Calibration Check and Operation

The turbidimeter has been calibrated by the manufacturer and the electronic calibration using freshly prepared formazin standards should only be performed if the electronic printed circuit board, the photodetectors or the light source has been replaced.

The procedures for calibration checks and the operation of the meter follows:

1. For accurate measurements in the low range rotate the cuvettes in the well to obtain the minimum reading. Mark the cuvette with one of the adhesive dots provided with the instrument so that orientation of the cuvette will be identical each time it is placed in the instrument.
2. To operate the turbidimeter, switch to the "10" range and place the Reference Standard (0.02 NTU) in the optical well.

3. With the light shield in place over the well, adjust the Reference Adjust knob to cause the meter to read the reference standard value on the scale. The unit is now ready for use in either range.

4. To make a measurement of a sample, clean one of the cuvettes and fill to within approximately 3/4" from the top with sample. Place the top on the cuvette and carefully clean the outside surface of the cuvette with a lint free wiper such as KimWipes. Place the sample in the well and place the light shield over the well. Select the appropriate range for best readability. Record results in field logbook.

5. Repeat steps 3 and 4 for each sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be within ±15%.

   If the results are outside of the control limits, clean cuvettes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below).

2. Calibration check results must be ±10% of the true value. If the result is outside of ±10%, clean cuvettes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in-control calibration.

3. All glassware is to be soap and water washed, tap rinsed and distilled or deionized water rinsed prior to analyses.

Interferences:

Interferences in turbidity measurements are generally due to dirty or scratched cuvettes. Handle only the top one-third of the cuvettes and wipe clean using a lint-free wiper (KimWipes or equivalent).

DISSOLVED OXYGEN

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, revised March 1983, Method 360.1

Sensitivity: 0.1 mg/L as O_2

Optimum Range: 0.1 mg/L to 20 mg/L O_2

Sample Handling: Determined on site

Reagents and Apparatus:

1. Temperature compensated dissolved oxygen (DO) meter, Corning Check Mate System;
2. Zero oxygen standard;
3. DO sensor-filling solution;
4. DO membrane replacement kit;
5. Distilled or deionized water in wash bottle.

Setting Up DO Sensor:

The sensor is shipped dry and must be filled before use.

1. Unscrew the membrane cap from sensor and fill using DO electrolyte.
2. Tap membrane cap gently to remove air bubbles. Gently screw cap onto probe body allowing surplus electrolyte to run out. (Caution: Do not overtighten)
3. Fit sensor to meter module.
4. Allow 30 minutes for polarization of electrode.

Calibration:

1. Remove wetting cap from tip of sensor. Switch on meter.

2. For first calibration point, place sensor in zero oxygen solution. Allow sufficient time for sensor to stabilize.

3. Move the sensor in a gentle circular motion.

4. Make sure sensor is immersed to a depth of 40 mm to cover the temperature-sensing element.

5. Press "CAL" key. CAL 1 is displayed on meter and after endpointing, the display automatically updates to zero.

6. For second calibration point, hold sensor in air. Press "CAL" key. CAL 2 is displayed. After endpointing, the display automatically updates to 100% O_2.

7. To adjust oxygen calibration for salinity and barometric pressure, press "Mode" key. In mg/L O_2 mode, press "CAL" key and 100 is displayed. Use down arrow and up arrow on the keypad to adjust the display according to the salinity and barometric pressure tables contained on the operating instruction leaflet.

Procedure:

1. Calibrate meter using calibration procedure.

2. Pour the sample into clean sample jar or plastic cup.

3. Place sensor in sample. After following the immersion, stirring and stabilization steps referred to during calibration, press "READ" key to obtain sample result.

4. Record result in the field logbook.

5. Repeat steps 2 through 4 for each sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be within 15%.

   If the results are outside of the control limits, rinse electrode and repeat analysis. If results are still outside of the control limits, recollect samples and repeat

analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If unable to recalibrate, replace sensor membrane.

2. Calibration check results must be within 10% of the true value. If the result is outside of 10%, rinse electrodes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in control calibration.

3. All glassware is to be soap and water washed, tap rinsed and distilled or deionized water rinsed prior to analyses unless pre-cleaned sample jars are used.

Interferences:

Interferences in DO measurements generally occur due to membrane coating. Clean probe as specified in the sensor manual.

The presence of other gases such as chlorine, nitrous and nitric oxide, hydrogen sulfide and sulfur dioxide interfere with DO measurements. The sulfur-based compounds will tarnish the electrodes resulting in sluggish or erratic measurements. Polishing the electrodes as specified in the operating manual will restore the performance of the meter. Recalibrate meter before analysis of next sample.

OXIDATION-REDUCTION POTENTIAL (ORP)

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Standard Methods for the Examination of Water and Wastewater", APHA, 18th edition, 1992, Method 2580B.

Sensitivity: 1 mV

Optimum Range: -1,500 to 1,500 mV

Sample Handling: Determined on site

Reagents and Apparatus:

1. ORP meter;
2. ORP electrode assembly;
3. Thermilinear thermistor temperature probe;
4. ZoBell Solution;
5. Distilled or deionized water in wash bottle.

Calibration:

1. Turn on the Water Quality Monitor and set the pH function switch to mV.
2. Connect the shorting cap attached to the to the mV input jack. The display should read 000-±2 mV. This indicates that the electronics are zeroed.
3. Detach the shorting cap and connect the to the mV input jack. If a pH electrode is not attached to the pH input jack, connect the shorting cap to it.
4. Attach the to the TEMP input jack.
5. Rinse with distilled or deionized water, followed by a rinse with a small amount of reconstituted ZoBell Solution.
6. Half fill a disposable 50 ml sample cup with ZoBell Solution and fully immerse the bulb and the end of the sheath. Allow the sensors to equilibrate, and note the reading.

7. The displayed mV values are not temperature compensated and should be corrected to 25°C at 1.3 mV/°C. The temperature coefficient is in reverse proportion to the temperature.

8. Correct the value to 25°C using the following equation:

   Actual Value mV = Display Value + [(Display Temp. - 25°C) x (1.3 mV)]

Procedure:

1. Calibrate meter using calibration procedure.

2. Set up meter as outlined in the instruction manual.

3. Record temperature and ORP of the sample in the field logbook.

4. Correct ORP to 25°C using the formula presented above.

5. Record corrected ORP in the field logbook.

6. Repeat steps 3 through 6 for each sample.

7. Recheck calibration with ZoBell solution after every ten or fewer samples and after the last sample.

8. Store electrode in soaker bottle when not in use.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be ±10 mV.

   If the results are outside of the control limits, rinse electrodes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If drift is suspected to be the cause of the problem, clean the electrode and recalibrate. If drift is still apparent, replace electrode.

2. Calibration check results must be 231±10 mV. If the result is outside of this range, rinse electrodes and check solution again. If still outside this range, recalibrate the meter and reanalyze all samples analyzed since the last in-control calibration.

Interferences:

Interferences in ORP measurements occur when the platinum electrode surface becomes coated. Clean the ORP electrode as follows:

1. Soft coatings should be removed by use of a wash bottle of water or by gently wiping with a soft cloth. Remove the bulb guard if necessary. Be careful not to scratch the platinum.

2. Hard coatings or organic chemicals should be removed by an appropriate chemical solvent, by gently scrubbing with a very fine cleansing powder such as "Softscrub", or by gently polishing with 600 grade wet silicon carbide paper. Wet a piece of the paper with water and gently polish the electrode with a circular twisting motion.

Note:
After cleaning the platinum surface, soak the electrode for an 8 to 24 hours in 4.0 pH buffer, and then recheck it with ZoBell Solution before further use.

SOIL VOC SCREENING

Scope and Application: This method is applicable to screening VOCs in the headspace of soil samples.

Method: Photoionization

Sensitivity: Approximately 0.5 ppm depending on background

Optimum Range: Background to 2,000 ppm

Sample Handling: Determined on site

Reagents and Apparatus:

1. HNu Photoionizer or Photovac Microtip Photoionization Detector;
2. Calibration gas Isobutylene 100ppm;
3. Calibration apparatus and tubing;
4. Battery charger.

Calibration:

A) Calibration of the HNu

1. Connect the analyzer to the regulator and calibration gas cylinder with a short segment (butt connection) of tubing. The calibration gas consists of a mixture of isobutylene and zero air. Isobutylene is non toxic and safe to use in confined areas. There are no listed exposure levels at any concentration.

   It is important that the tubing be clean since contaminated tubing will affect the calibration reading. A cylinder containing less than 30 psig pressure will not be used as readings below that level can deviate up to ten percent from the rated value.

   Turn the function switch to the STANDBY position.

2. With the SPAN setting and the function switch at the same positions as listed in the Application Data Sheet or Calibration Report, open the valve on the cylinder until a steady reading is obtained.

3. If the reading is the same as the recorded data, the analyzer calibration for the original species of interest is still correct.

4. If the reading has changed, adjust the SPAN setting until the reading is the same.

5. Shut off the cylinder as soon as the reading is established.

6. Record and maintain this new SPAN setting.

7. Background organic vapor readings should be measured prior to any soil screening.

B) Calibration of MicroTip

1. Connect the supplied regulator to the span gas cylinder. Hand tighten the fittings. Observe proper handling techniques for all gases.

2. Open the valve on the gas bag by turning the valve stem fully counterclockwise.

3. Attach the nut to the regulator. Hand tighten the fittings.

4. Turn the regulator knob counterclockwise about half a turn to start the flow of gas.

5. Fill the gas bag about half full and then close the regulator fully clockwise to turn off the flow of gas.

6. Disconnect the bag from the adapter and empty it. Flush the bag a few times with the span gas and then fill it.

7. Close the gas bag by turning the valve clockwise.

8. Press SETUP and select the desired Cal Memory with the arrow keys and press ENTER. Press EXIT to leave Setup.

9. Press CAL and enter the desired response factor. Use Table 2 from the manual to find the correct response factor. If the compound is not in

Table 2 or you are not looking specifically for one compound then enter 1.00.

The concentration detected by MicroTIP will be multiplied by the response factor before it is displayed and logged.

10. Expose MicroTIP to zero air. Press ENTER and MicroTIP sets its zero point.

11. MicroTIP then asks for the span gas concentration. Enter the known span gas concentration and then connect the span gas bag adapter to the inlet.

12. Press ENTER and MicroTIP sets its sensitivity.

13. When MicroTIP's display reverts to normal, MicroTIP is calibrated and ready for use. Remove the span gas bag from the inlet.

Procedure:

1. Calibrate meter using the correct calibration procedure for the meter used.

2. The samples for VOC headspace screening will be prepared in the field by filling a 4-ounce soil jar to one-half its volume and sealing with a teflon-lined closure. The remaining sample will be placed in the appropriate jars for the analyses required.

3. Allow the sample for VOC headspace screening to remain at ambient temperature for a minimum of 10 minutes. This will allow for VOCs in the soil to reach equilibrium in the headspace of the jar.

4. Remove the lid of the soil jar slightly and insert the probe of the meter into the headspace.

5. Take the highest reading from the meter or readout of the instrument.

6. Record the reading in the field logbook.

7. Recheck calibration with calibration gas after a minimum of every 10 samples and after the last sample.

Quality Control:

1. Calibration check results must be ±10 percent of the true value. If the result is outside of ±10 percent, recalibrate the meter as specified above.

2. Duplicate samples are not analyzed since the headspace VOC readings will vary considerably as the soil VOCs volatilize.

Interferences and Limitations:

Humid conditions will cause a negative bias to the reading. The photoionization detection principle used by the instruments will not detect all VOCs. The instrument is most sensitive to aromatic, alkene and alkyne VOCs.

FIELD PROCEDURES FOR MEASURING IRON II (FERROUS)

Scope and Application: This method is applicable to screening Iron II (Ferrous) in groundwater.

Method: Powder Pillows or AccuVac Ampuls

Summary of Method: The 1,10-phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration.

Sensitivity: The estimated detection limit is 0.03 mg/L

Sampling and Storage: Ferrous iron must be analyzed immediately and cannot be stored. Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not measured.

Reagents & Apparatus - Using Powder Pillows:

Description	*Quantity Required Per Test*	*Units*	*Cat. No.*
Ferrous Iron Reagent Powder Pillows	1 pillow	100/pkg	1037-69
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

Reagents & Apparatus - Using AccuVac Ampulls:

Description	*Quantity Required Per Test*	*Units*	*Cat. No.*
Ferrous Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25140-25
Beaker, 50 mL	1	each	500-41H

Calibration

1. No field calibration

Procedure - Using Powder Pillows

1. Enter the stored program number for Ferrous iron (Fe^{2+})- powder pillows.
2. Press: **PRGM** The display will show: **PRGM ?** ***Note:*** *Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not determined.*
3. Press: 33 ENTER; the display will show mg/L, Fe and the ZERO icon.
4. Fill a sample cell with 25 mL of sample (the blank).
5. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.
6. Press: **ZERO**; The cursor will move to the right, then the display will show: **0.00 mg/L Fe**
7. Fill another sample cell with 25 mL of sample.
8. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to mix. ***Note:*** *Undissolved powder does not affect accuracy.*
9. Press: **TIMER ENTER**; A three-minute reaction period will begin. ***Note:*** *An orange color will form if ferrous iron is present.*
10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.
11. Press: **READ**; The cursor will move to the right, then the result in mg/L ferrous iron will be displayed. ***Note:*** *Standard Adjust may be performed using a prepared standard (see Section 1).*

Procedure - AccuVac Ampuls

1. Enter the stored program number for ferrous iron (Fe^{2+}) AccuVac ampuls.
2. Press: **PRGM**; The display will show: **PRGM ?** **Note:** Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined.
3. Press: **33 ENTER**; the display will show **mg/L, Fe** and the **ZERO** icon.
4. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.
5. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.
6. Press: **ZERO**; the cursor will move to the right, then the display will show: **0.00 mg/L Fe**
7. Fill a Ferrous Iron AccuVac Ampul with sample. **Note:** Keep the tip immersed while the ampul fills completely.
8. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints. **Note:** undissolved powder does not affect accuracy.
9. Press: **TIMER ENTER**; a three-minute reaction period will begin. **Note:** An orange color will form if ferrous iron is present.
10. Place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.
11. Press: **READ**; the cursor will move to the right, then the result in mg/L ferrous iron will be displayed. **Note:** Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. NC-SC-0005, Rev. 6.4
Effective Date: 10/02/06
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Title: Sample Receiving and Sample Control

[Method: None]

Approvals (Signature/Date):

Technology Specialist	12/14/07 Date	Health & Safety Coordinator	12-14-07 Date
Quality Assurance Manager	12/13/07 Date	Laboratory Director	12/14/07 Date

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STL STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIVING AND SAMPLE CONTROL

(SUPERSEDES: REVISION 6.3, 04/20/04)

Reviewed by:	Technology Specialist	12/4/06 Date
Approved by:	Quality Assurance Manager	10/4/06 Date
Approved by:	Environmental Health and Safety Coordinator	10-4-06 Date
Approved by:	Laboratory Director	12/5/06 Date

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1. SCOPE AND APPLICATION

1.1. It is the responsibility of Sample Receiving and Control personnel to perform the procedures described herein in full compliance with this SOP.

1.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable personnel to perform this SOP correctly.

1.3. Analyst

1.3.1. It is the responsibility of the analyst to provide the correct request(s) for bottles to the sample custodian by using the QuantIMS program, PSR024.01 (as described in section 11.25) and to return all bottles to the custodian.

1.3.2. It is the responsibility of the analyst or designee to fill all bottle requests in a timely fashion and document the transfers on the request/return forms (Appendix 17.2.5).

1.3.3. It is the responsibility of the analyst or designee to correctly document the bottle information required (Appendix 17.2.5).

1.4. Sample Custodian

1.4.1. It is the responsibility of the sample custodian or designee to ensure that the returned bottle is the same as the one relinquished and to return it to the proper storage area.

1.4.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. Not applicable.

3. DEFINITIONS

3.1. Sample Custodian refers to sample control personnel or designee.

3.2. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version

4. INTERFERENCES

4.1. Not applicable.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn when unpacking coolers, aliquoting total solids samples, purging samples, and any other task that presents a strong possibility of getting cut. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. 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/Unusual Hazards
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.
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.
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.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
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.			

5.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported immediately to the EH&S Coordinator and the Laboratory Supervisor.

5.7. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

6. EQUIPMENT AND SUPPLIES

6.1. Thermometers

6.2. PPE such as gloves, lab coats, safety glasses, etc.

6.3. Utility knives

6.4. pH paper

6.5. Copier, printer, computer and label generator

6.6. Carts

7. REAGENTS AND STANDARDS

7.1. Not applicable.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Not applicable.

9. QUALITY CONTROL

9.1. Nonconformance and Corrective Action

9.1.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable.

11. PROCEDURE

11.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

11.2. The procedures listed in this document describe the responsibilities of Sample Control personnel in ensuring that data is transmitted correctly from the client samples to all personnel involved with sample analysis and review.

11.3. The intent of the sample custodian maintenance program is to show custody of individual bottles based on work order number as well as bottle control numbers. This program acts as an internal chain of custody.

11.4. The sample control group opens each cooler and removes the enclosed sample documents. Coolers are prioritized based on rush status and expirable tests. If a COC (chain-of-custody) is marked rush, the following occurs:

24 hour TAT – flag with red folder
48 hour TAT – flag with blue folder
72 hour TAT – flag with yellow folder
1 week TAT – flag with green folder

11.5. If tests on the COC are expirable, the cooler is marked with a manila folder.

11.6. Rush and expirable coolers are to be unpacked first and logged as soon as possible. The lab groups are to be notified with any rush 72 hours or less.

11.7. The following information is documented on the Cooler Receipt/ Narrative Form (Appendix 17.2.1).

11.7.1. Samples were received via overnight courier, client drop off, or other means.

11.7.2. Presence of the custody seals on the outside of the cooler

11.7.3. Presence of the custody papers.

11.7.4. The custody papers were properly filled out (ink, signed, match labels)

11.7.5. The custody papers were signed in the appropriate place

11.7.6. Presence of the shipper's packing slip

11.7.7. Presence of packing material information: if yes, type of packing material

11.7.8. The temperature of the cooler is taken by one of the following methods that best reflect the condition/temperature of the samples upon receipt: temp vial, coolant/sample, between bottles, IR, Ice/H_2O slurry.

11.7.8.1. If temp vial is present, the temperature of the temp vial is taken as soon as it is removed from the cooler. A temperature probe is inserted into the temp vial to obtain the temperature.

11.7.8.2. In the use of the coolant/sample methods, the temp is taken by placing the thermometer probe between the coolant and the sample(s).

11.7.8.3. In the use of between bottles method, the thermometer probe is placed between two sample bottles and the temperature recorded.

11.7.8.4. The IR gun is used on a single bottle, sometimes the temp blank, that best reflects the cooler temperature.

11.7.8.5. If the Ice/H_2O slurry method is used, the temperature is taken from the ice/slurry mixture. This method is only used if all sample bottles are in contact with the slurry.

11.7.8.6. If the temperature is outside 4°C ± 2°C, the anomaly is recorded on the cooler receipt form. The project manager is contacted when the temperature is >6°C.

11.7.9. Condition of bottles upon receipt (good condition, broken, etc.)

11.7.10. Complete bottle labels (date, time, client ID)

11.7.11. Information on bottle labels and tags agree with custody papers

11.7.12. Correct bottles used for the tests indicated

11.7.13. VOA bottles were checked for the presence of air bubbles. Any bubbles exceeding 6 mm in diameter are narrated and the PM is contacted.

11.7.14. Sufficient amount of sample sent in each bottle

11.7.15. pH's are taken, on all preserved samples less Volatiles, TOC, and TOX by removing sample lids and using a droplet of sample from in the lid to test the pH. The pH's are then recorded on the cooler receipt form. The pH paper strips are then discarded.

11.7.15.1. In determining if the sample is preserved to the correct pH for Navy samples, the sample custodian or receiving personnel must take an aliquot of sample out of the sample container either by pouring out a small aliquot or using a disposable pasteur pipette and place a drop of the sample on a pH paper strip.

11.7.16. Purchased prepared vials of preservatives are used if samples are not at the correct pH. The pH is adjusted by adding the appropriate preservative in 5 mL increments up to a maximum of 20 mL per liter of sample or unless there is a reaction. Sulfides are preserved with 5 mL Sodium hydroxide and 1 mL Zinc acetate. The pH adjustment and final pH are noted on the cooler receipt form. The Lot Number of the pre-made preservative is located on the Cooler Receipt Form. It is the responsibility of the Sample Receiving Group to change the lot number when a new shipment arrives.

11.7.17. The concentrations of the preservatives used:

4N Sodium Hydroxide
1N Zn Acetate
1:1 HCL (18%)
1:4 $HN0_3$ (18%)
1:2 H_2SO_4 (33%)

11.7.18. If the Project Manager was notified of any discrepancy/non-conformance at log-in, the information is recorded on cooler receipt form with the name of the Project Manager, date contacted, name of sample custodian who contacted the Project Manager, and how contacted.

11.8. The Sample Control person is to remove all sample containers. Any broken, leaking, or dirty sample containers are to be placed inside the fume hood. Dirty sample containers are to be cleaned appropriately, so as not to contaminate the sample storage area. The Sample Control person is to wear disposable latex gloves, safety glasses, and a lab coat while handling any samples.

11.9. Any cooler received emitting strong vapors/fumes when opened will be taken to the High Hazard Room and unpacked in either of its hoods.

11.9.1. Any problems concerning exposure while unpacking samples must be immediately reported to the Group Leader or Supervisor.

11.10. Any volatile sample(s) suspected (e.g., odor) or known (client information or site history) to be high in volatile concentration is stored in a separate designated volatile area.

11.11. The Sample Control person is to examine all documents and compare information from sample container labels and Chain-of-Custody Records to insure that there is no discrepancy between documents, ensuring that all documents are properly completed and signed.

11.12. If any problems or discrepancies are noted during the sample receiving process that compromise sample integrity, such as limited sample volume, sample identification cannot be determined from the COC, incorrect pH levels (or preservatives if known), or broken, leaking samples, the Project Manager is notified. They in turn will contact the client in an attempt to solve discrepancies.

11.13. Expirable tests (hold time 48 hours or less) must be written on the top of the bottles from which they are to be analyzed. If more than one method exists for the analysis, the method must also be written.

11.14. Any sample requiring a Total Solid result is split off for analysis. Splitting these samples is the responsibility of the receiving group. A small representative portion (approx. 5-10 grams) from each sample is put into a small plastic snap-top container designated for the TS analysis. If the TS container is not labeled with a QuantIMS label at the time of splitting off, the TS container must be labeled with handwritten client ID. Containers designed for VOC analysis must not be opened/used for TS aliquot. When only one solid container is received for VOC analysis, receiving group must **not** split off because of possible contamination or possible lost volatiles. An empty TS container with a QuantIMS label is given to the VOC analysis group for each solid. The VOC group will aliquot for the TS when they open container for analysis. TS plastic containers are placed into baggies by lot (project) and the baggies are put into a box inside the walk-in cooler door.

11.15. When samples need to be composited, the following procedure is followed, unless there are specific instructions from the client.

11.15.1. Equal aliquots are weighed from each container and mixed thoroughly and transferred to a new container.

11.15.2. The amount aliquoted is recorded on the Cooler Receipt Form.

11.16. If all samples recorded on the Chain-of-Custody Record were received by the laboratory and there are no problems observed with the sample shipment, the Sample Control person will sign the Chain-of-Custody Record in the "Received for Laboratory by:" box on the document. If problems are noted, sign for shipment and note the problems. All discrepancies are recorded on Cooler Receipt Form.

11.17. A quote must reflect what is on the chain-of-custody. Any discrepancies must be resolved by the Project Manager. Likewise, if there is not an associated quote in QuantIMs, the samples are placed on hold until a quote is completed by the Project Manager.

11.18. In the event that a project is on hold until the next day or longer, all associated paperwork is placed into a black folder and all samples and black folder are put into cold storage. Any project that is on hold must be recorded on the dry-erase board posted on walk-in cooler door.

11.19. The Sample Control person will enter each sample into the laboratory computer (QuantIMS), where a unique lot number is assigned to each project received, and sequential sample numbers are designated for each client identification within the lot.

11.19.1. Lot Numbers: The lot number is nine characters in length and is based on the date of receipt. Lot number A9J010121 is described as follows:

A - STL location where the samples were received.

(A = North Canton, B = Tampa, C = Pittsburgh, etc.)

9 - Last digit of the year (i.e. 1999).

J - Month (i.e. A = January, B = February, J = October, etc.)

01 - The next 2 numeric characters identify the day of the month, in this case, the first day of the month.

0121 - The next 4 numeric characters are the sequential assignment of numbers specific to each lot received. Each day the first lot logged in receives the number "0101", the second lot receives the number "0102", etc..

For example:

If four bottles were submitted under Client ID numbers AB100-AB103 and the laboratory identification number generated by the computer is A9K100101, then the assigned laboratory number recorded on the Sample Log-In Sheet would be as follows.

Client ID Sample Number	Assigned Laboratory Number
AB 100	A9K100101-001
AB 101	A9K100101-002
AB 102	A9K100101-003
AB 103	A9K100101-004

11.19.2. Sample Numbers: The samples in each lot are assigned a sample number that is attached to the lot number and are reset at each new lot. For example: the first and second samples in the lot above are labeled A9J010121-001 and A9J010121-002.

11.19.3. Sample Suffixes: Each sample also has a 1 character field (which is not a required field for all samples) called the suffix which identifies the sample as specified below.

Client Sample	no suffix
Method Blank	B
Laboratory Control Sample	C
Laboratory Control Sample Duplicate	L
Matrix Spike	S
Matrix Spike Duplicate	D
Sample Duplicate	X
Serial Dilution	P
Sample Confirmation	Y
Post Digestion Spike	Z
Re-analysis	I

Example: A9J010121-001X is a sample duplicate for sample A9J010121-001.

11.19.4. Work Order Numbers: Each test requested by the client for an individual sample receives an individual 8 digit work order number assigned by QuantIMS. Work order number A5WE1-2-1C is described as follows:

A5WE1 - In addition to the three digit sample identification described in 4.7.2 (i.e. - 001 and - 002), the first 5 characters of the work order number also identifies each unique sample. This identification is generated in QuantIMS using a sequential logic that is beyond the scope of this SOP to describe.

2 - The "modifier" indicates the type of run. In this case this is the second time the sample had to be run. If it needs reprepped and run again, the number would indicate a "3". The original analysis work order number assigns "1" to the modifier position.

1C - The "suffix" is the identification of the specific test for that sample. The suffix in this case is not always sequential, but is unique to the test to be performed on the sample.

Example: A5WE1-2-1C is the assigned 8 digit work order number for the reanalysis of the chloride test on the sample A5WE1. A5WE1-1-05 could be the 8 digit work order number for the analysis of SW846 8270 on sample A5WE1.

11.19.5. Each sample container with the same client ID has a unique number. Each container will be labeled with the same 5 digit work order number and then will contain a suffix beginning with –001, -002, -003, etc. For example, if 5 containers are submitted from the same sample point and the LIMS-generated work order number is CREE4, LIMS will generate 5 labels: CREE4-001, CREE4-002, CREE4-003, CREE4-004, CREE4-005. The specific bottle number is the number used for sample request/removal paperwork.

11.19.6. Labels that read "Caution-Use Hood!" shall be affixed to all containers for a given sample that are thought to be a safety hazard (for example, high in contaminants, flammable, etc.), or known to emit noxious odors (this includes all DuPont samples). The Sample Receiving group is notified of potential hazards by the Project Manager, COC, quote, or client.

11.19.6.1. Samples that are known or expected to contain high concentration of Cyanide (250 ppm or more) or Sulfide (500 ppm or more) need to be unpacked in a fume hood. The Sample Control Group must put a special sticker on these sample bottles indicating to the Lab Groups that the samples are high in either Cyanide or Sulfide so the Lab Groups can take the necessary safety precautions.

11.19.7. Expirable tests must be given to the lab groups the day they are received. The expirable test/method is written on top of the bottle and the bottle must be put in the red bin designated for expirables. The work order or sample ID must be recorded on the expirable logsheet along with the record of the test to be run, special method if necessary and the initials of the person relinquishing the sample. The Wet Chem lab group checks this bin throughout the day and is responsible for signing out the sample container when they take it.

11.19.8. Once all sample containers have been properly labeled and all the information has been recorded on the Sample Lot Summary; the Sample Control person will place the samples into the proper storage locations. These locations are as follows:

11.19.9. Organic extractable samples (Semivolatiles, Pesticides/PCBs) are to be placed into the walk-in refrigerators located in Sample Receiving.

11.19.10. Volatile samples are to be stored in the double-door refrigerator located in the Sample Custodian area.

11.19.11. Samples known or suspected to be of high concentration are not stored in these refrigerators located in Sample Receiving.

11.19.12. Inorganic samples are to be placed into the walk-in refrigerators located in Sample Receiving.

11.19.13. Preserved metal samples are placed in a non-refrigerated room located in the Sample Custodian area. Metals samples that need to be lab filtered and/or preserved are stored in the walk-in cooler. All metals for Navy projects are stored in the walk-in cooler.

11.20. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.21. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.21.1. For clients who request a show of sample transfer from sample receipt to storage, a sample control record is printed. (see Appendix 17.2.4.). STL LIMS will generate this record for Expanded Deliverable and CLP designated samples. This record is referred to as an internal chain of custody (COC). This form can also be generated from the STL North Canton website.

11.22. The completed sample control record is attached to the summary package. The sample control record can be manually printed using the SAM S31 command in QuantIMS. Note: The report package for the lot must be "D" (Expanded Deliverable) or "C" (CLP) for the report to print.

11.23. Samples received after hours are signed for by an STL North Canton employee and placed in the walk-in cooler to be processed the following business morning.

11.24. Samples are requested by an analyst through the QuantIMS program PSR024.01. This program is accessed from the Sample Receiving Menu (SAM) by selecting option S24 (Sample Removal Requests). Bottles are requested based on method code, workorder number, prep code, QA batch number, lot number, or method group.

11.25. Each bottle has an associated lot number, sample workorder number, and bottle suffix number (Appendix 17.2.7).

11.26. When an analyst requests bottles and exits the request program, a requisition (Appendix 17.2.5) prints in the custodian's work area. This requisition identifies the requestor and the method/parameter requested. The requestor fills the requisition, recording each bottle number on the form. The bottle(s)

is/are signed out to the custody of the requestor by indicating the name and the date of transfer. The request is relinquished and accepted by the requestor's initials.

11.27. When bottles are returned, the sample custodian records the return on the original requisition form and re-enters custody of the bottles to the sample control area. The custodian initials and dates the return on the form (Appendix 17.2.5).

11.28. When sample bottles are consumed in the analysis process, the empty container is returned to the custodian. The custodian marks a "C" by the appropriate sample on the request form to indicate a consumed sample (Appendix 17.2.5).

11.29. Samples returned after the custodian has left at the end of the day are placed in the walk-in cooler by a custodian designee and recorded as received by the custodian the next working day.

11.29.1. All request forms remain in the sample custodian area while samples are being analyzed. The final form is also kept in the sample custodian area after samples have been analyzed and reported.

11.29.2. When a sample custodian is not available, a designee gets the samples, and all request paperwork is completed properly.

11.30. Subcontracting of samples

11.30.1. Samples that are logged but not analyzed at the laboratory are subcontracted to different laboratories for analysis including other STL facilities.

11.30.2. The LIMs system will automatically print a Sample Analysis Requisition for these samples upon completion of the log-in process (Appendix 17.2.6).

11.30.3. This form contains information necessary for sample analysis. The original form is sent to the subcontracted laboratory and a copy is attached to the summary package. The Sample Analysis Requisition form must have a relinquished signature with a date and time. Any additional information necessary for sample analysis must be handwritten on the form (e.g. list of compounds, homogenizing of samples, limited quantity, etc.). In order to track subcontracted samples, the lab purchase order number on the Sample Analysis Requisition form must be recorded in the subcontracted sample PO book located in the receiving log-in area. (Appendix 17.2.7).

11.30.4. A sample analysis request (SAR) can be printed from LIMS or the STL North Canton website by entering the Lot Number.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable.

13. METHOD PERFORMANCE

13.1. Training Qualifications:

13.1.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.1.2. The only personnel authorized to execute this SOP are the Sample Log-In persons.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, Sate, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety manual for "Waste Management and Pollution Prevention".

15.2. All samples may be disposed of 30 days after the report date except for those samples associated with special client retention. Shelves are purged in chronological order. All lots on a specific shelf must be noted. If a lot can be disposed of, a disposal date must be recorded.

15.3. Disposal dates are recorded on a print-out of lots according to a storage location. When clearing samples that cannot yet be disposed of because of special client retention, samples are boxed or stored on carts. Stored samples must have a specific date listed in which samples can be disposed of, or a note that indicates "SAVE" and client name or reason.

15.4. All lots assigned to a certain shelf can be obtained from QuantIMS.

15.4.1. UTL – "Enter"

15.4.2. Type "wrk" – "Enter"

15.4.3. Type "2" by option

15.4.4. Choose one query name and "Enter"

15.4.5. SLOIS for metals shelves

15.4.6. CLOIS for cooler shelves C1 – C272

15.4.7. WLOIS for cooler shelves W1-163

15.4.8. Type "1" next to "select records"

15.4.9. Enter the shelf location 3x where prompted (appears on two different screens). Then hit "Enter".

15.4.10. Dates need to be updated every four to six months so shelf locations only print most recent lots assigned.

15.4.11. "F3" to run query and "Enter".

15.4.12. At save definition type "Y" and run option "2" and "Enter".

15.5. Solid samples that are non-regulated waste are placed in a cubic yard container for disposal.

15.6. Regulated solid waste is placed in the "MIXED WASTE" container.

15.7. Water samples designated for disposal are placed on carts and disposed of in one of the following areas: sample receiving, wet chemistry, or extractions. Acidified samples are poured into a drum and neutralized as close to a fume hood as possible in one of the following areas: sample receiving, wet chemistry, or extractions.

15.8. Solvent waste must be disposed of in clearly labeled waste cans.

15.9. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1 STL Quality Management Plan (QMP), current version.

16.1.1 STL Laboratory Quality Manual (LQM), current version

16.1.2 Corporate Quality Management Plan (QMP), current version

16.1.3 STL Corporate Safety Manual, M-E-0001, and STL North Canton Facility Addendum and Contingency Plan, current version

16.2 Associated SOPs and Policies, latest version

16.2.1 QA Policy, QA-003

16.2.2 Navy/Army SOP, NC-QA-0016

17 MISCELLANEOUS

17.1 Wherever "Sample Control" is mentioned in all SOPs, it is assumed to include the sample custodian or any alternate that is designated by the Sample Control Coordinator.

17.2 Appendices

17.2.1 Appendix I – Cooler Receipt Form/Narrative

17.2.2 Appendix II - Preservative Preparation

17.2.3 Appendix III - Preservative Requirements

17.2.4 Appendix IV – Sample of Internal Chain of Custody

17.2.5 Appendix V - Sample of Custodian Removal Request

17.2.6 Appendix VI – Sample of Client Analysis Summary

17.2.7 Appendix VII - Laboratory Generated Bottle Label

Appendix I - Cooler Receipt/Narrative Form

STL Cooler Receipt Form/Narrative Lot Number:_____

North Canton Facility

Client:_____ Project:_____ Quote#:_____

Cooler Received on:_____ Opened on:_____ by: _____

(Signature)

Fedx ☐ Client Drop Off ☐ UPS ☐ Airborne ☐ FAS ☐ Other:_____

Cooler ☐ Safe ☐ Foam Box ☐ Client Cooler ☐ Other:_____

STL Cooler No#:_____

1. Were custody seals on the outside of the cooler? Yes ☐ No ☐ Intact? Yes ☐ No ☐ NA ☐

   If YES, Quantity_____

   Were the custody seals signed and dated? Yes ☐ No ☐ NA ☐

2. Shipper's packing slip attached to this form? Yes ☐ No ☐ NA ☐

3. Were custody papers included inside the cooler and relinquished? Yes ☐ No ☐

4. Did you sign the custody papers in the appropriate place? Yes ☐ No ☐

5. Packing material used:

Peanuts ☐ Bubble Wrap ☐ Vermiculite ☐ Foam ☐ None ☐ Other :_____

6. Cooler temperature upon receipt _____ °C (see back of form for multiple coolers/temp)

METHOD: Temp Vial ☐ Coolant & Sample ☐ Against Bottles ☐ IR ☐ ICE/H_2O Slurry ☐

COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ Water ☐ None ☐

7. Did all bottles arrive in good condition (Unbroken)? Yes ☐ No ☐

8. Did all bottle labels and tags agree with the custody papers? Yes ☐ No ☐

9. Were samples at the correct pH? (record on back) Yes ☐ No ☐ NA ☐

10. Were correct bottles used for the tests indicated? Yes ☐ No ☐

11. Were air bubbles >6 mm in any VOA vials? Yes ☐ No ☐ NA ☐

12. Was a sufficient amount of sample sent in each bottle? Yes ☐ No ☐

Contacted PM _____ Date:_____ by:_____ via Voice Mail ☐ Verbal ☐ Other ☐

Concerning:_____

√	MACRO	MACRO

1. CHAIN OF CUSTODY

	SR1A	The chain of custody and sample bottles did not agree. The following discrepancies occurred_____

2. SAMPLE CONDITION

	SR2A	Sample(s)_____ were received or requested after the recommended holding time had expired.
	SR2B	Sample(s) _____ were received with insufficient volume.
	SR2C	Sample(s) _____ were received in a broken container.

3. SAMPLE PRESERVATION

	SR3A	Sample(s) _____ were further preserved in sample receiving to meet recommended pH level(s). *Nitric Acid Lot #120503-HNO3; Sulfuric Acid Lot # 101503-H2SO4; Sodium Hydroxide Lot # 111401-NaOH; Hydrochloric Acid Lot # 100902-HCl; Sodium Hydroxide and Zinc Acetate Lot # 112801-CH3COO2ZN/NaOH*
	SR3B	Sample(s)_____were received with bubble > 6 mm in diameter (cc: PM)

4. Other (see below or back)

Appendix II - Preservative Preparation

Preservative Preparation

(If purchased preservative solution vials are not used)

1:1 Hydrochloric Acid (18%): Slowly add 1000 mL concentrated HCl to 1000 mL reagent water and mix. Store in a well-labeled plastic coated acid bottle.

1:2 Sulfuric Acid (33%): In a 2000 mL beaker, SLOWLY and CAREFULLY add 500 mL concentrated H_2SO_4 to 1000 mL reagent water and mix. A cool water bath may be needed to cool the solution and beaker. Store in a well labeled plastic acid bottle.

NOTE: *All preparations must be performed in a hood and proper personal protective equipment must be worn. All reagents and final preservative solution must be documented in applicable reagent logbooks.*

Appendix III - Preservative Requirements

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
Asbestos	P	None	250 mL	Radiological Alpha, Beta, Radium	P	HNO_3	4 L
Acidity	P	None	250 mL	Hardness	P	HNO_3	250 mL
Alkalinity (Sep)	P	None	250 mL	Metals	P	HNO_3	1 L
BOD	P	None	250 mL	Dissolved Metals*	P	HNO_3	1 L
Carbonaceous BOD	P	None	250 mL	Total Organic Carbon (TOC)	G	HCl	2 x40 mL
Bromide (Br)	P	None	250 mL	Chemical Oxygen Demand	P	H_2SO_4	250 mL
Chloride (Cl)	P	None	250 mL	Total Organic Halogens	G	H_2SO_4	250 mL
Chromium, $^{6+}$	P	None	250 mL	COD	P	H_2SO_4	250 mL
R. Chlorine	P	None	100 mL	Ammonia Nitrogen (NH_3)	P	H_2SO_4	500 mL
Color	P	None	50 mL	TKN	P	H_2SO_4	1L
Conductivity	P	None	250 mL	Nitrate/Nitrite	P	H_2SO_4	250 mL
Corrosivity	P	None	250 mL	Oil & Grease	G	H_2SO_4	1 L
Dissolved Oxygen	G	None	300 mL	Phenols	G	H_2SO_4	1 L
Fecal Coliform	P	None	125 mL	Total Phosphorus	P	H_2SO_4	250 mL
Flashpoint	G	None	100 mL	TON	P	H_2SO_4	1 L
Fluoride	P	None	250 mL				
Nitrate	P	None	250 mL	TRPH - IR 418.1	G	HCl	2 L
Nitrite	P	None	250 mL	VOC 601	G	HCl	3x40 mL
pH	P	None	50 mL	VOC 8010	G	HCl	3x40 mL
Elemental PO_4	G	None	250 mL	VOC 624	G	HCl	8x40 mL
Orthophosphate	P	None	250 mL	BTEX 8020	G	HCl	3x40 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
TDS	P	None	250 mL	VOC 8240	G	HCl	3x40 mL
TSS	P	None	250 mL	THM/502.2	G	HCl	2x40 mL
Total Solids	P	None	250 mL	502.2	G	HCl & Asc. Acid	2x40 mL
TVS	P	None	250 mL	VOC 624	G	HCl	3x40 mL
T. Coliform	P	None	125 mL	VOC 602	G	HCl	3x40 mL
Settleable Solids	P	None	1L	465 C & D	G	HCl	4x40 mL
Silica	P	None	250 mL	BTEX 8021	G	HCl	3x40 mL
Sulfate	P	None	250 mL	VOC	G	HCl	3x40 mL
Sulfite	P	None	250 mL	VOC 8260	G	HCl	3x40 mL
Surfactants (MBAS)	P	None	250 mL	VOC and VOA	G	HCl	3x40 mL
Turbidity	P	None	250 mL	VOC 8010/8020	G	HCl	3x40 mL
TPH-GC	G	None	2 L				
				Total Cyanide	P	NaOH[3]	250 mL
BNAs	G	None	2 L	Amenable Cyanide	P	NaOH	250 mL
BNA + Dioxin	G	None	2 L	Free Cyanide	P	NaOH	250 mL
PNA/PAH	G	None	2 L	Sulfide	P	Zn Acetate & NaOH	1 L
Pesticides	G	None	2 L	Formaldehyde	G	None	500 mL
Reactive Cyanide	P	None	1 L	Carbonate	P	None	250 mL
Reactive Sulfide	P	None	1 L	Bicarbonate	P	None	250 mL
PCB	G	None	2 L	TPH - Diesel (Ext.)	G	None	2 L
Pesticides + PCBs	G	None	2 L	TPH - Gasoline (P&T)	G	HCl	2x40 mL
Herbicides	G	None	2 L	Glycols 8015	G	None	2x40 mL
OPPs	G	None	2 L	BTEX & MTBE	G	HCl	3x40 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
				601/602	G	HCl	3x40 mL

* Filtered in field

[1] HCl, HNO_3, and H_2SO_4 to pH < 2. NaOH to pH > 12

[2] Temperature = 4°C ± 2°C except for aqueous metals

[3] Samples to be analyzed for Cyanide should be field-filtered for Residual Chlorine. If Residual Chlorine is detected, ascorbic acid (0.6 g) should be added.

Appendix IV – Sample Internal Chain of Custody

	Lot: A4B180167 Quote: 49535						
	Project Number: 4500068724						
	PO Number:						
	Site: WOOSTER NWF/NPDES OUTFALL						
	Contact: Rich Kuhn Received: 02/18/04 12:10						
	Analytical Due Date: 02/25/04						
	Report Due Date: 03/03/04						
0	Client Sample ID	Date	Time	WA TER	MS 8260 LL	PH LIQ	A4B180167
1	217	2/17/04	10:00	WG	X	X	REPORT QC. NPDES. 1 WEEK TAT.

Appendix V - Sample Custodian Removal Request Form (LIMS Generated)

PSR024 2/18/04 16:08:32 MT SAMPLE CUSTODIAN REMOVAL REQUEST PAGE 001

REQUESTED BY: GIRARDS2

METHOD: C8 Fluoride (300.0, Ion Chromatography)

STORAGE LOCATION	PICKED WORK ORDER #	CNTR#	CONTROL #	CLIENT #	MATRIX ANALYSIS	LOTID	SMP#	SFX	MATRIX DESCRIPTION	QTY RCVD	QTY REQD
None	F8KPP-1-AL	___	931601	001628	I-88-C8 A4A020103	003			WATER	1	1
None	F8KPQ-1-AL	___	931602	001628	I-88-C8 A4A020103	004			WATER	1	1
None	F8KPT-1-AL	___	931603	001628	I-88-C8 A4A020103	005			WATER	1	1
None	F8KPV-1-AL	___	931604	001628	I-88-C8 A4A020103	006			WATER	1	1

13. RELINQUISHED BY DATE/TIME	RECEIVED BY	

***** END OF REPORT *****

Appendix VI - Sample Analysis Requisition

Severn Trent Laboratories, Inc.
Laboratory SAMPLE ANALYSIS Report
Report
REQUISTION

Package:

STL Valparaiso

2400 CUMBERLAND DRIVE
Valparaiso, IN 46383

STL Valparaiso Lab RequestSR056490Need Analytical Report
2004-02-24

Project Manager:
Client Code:

Sample I.D. Analysis Required	Work Order Number	Client Sample ID	Sampling Date
A4B100272-1	F9DED	MW-65A	2004-02-04
A4B100272-2	F9DEK	MW-66A	2004-02-05

Appendix VII - Laboratory Generated Bottle Label

F9P8N-001 3543
A4B180106-001 2/18/04
2XL/1X500ML 7:30
P1-02/18/04-GRAB
SW46

SF9P8N-002 3543
A4B180106-001 2/18/04
2XL/1X500ML 7:30
P1-02/18/04-GRAB
SW46

F9P8N-003 3543
A4B180106-001 2/18/04
2XL/1X500ML 7:30
P1-02/18/04-GRAB
SW46

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. NC-QA-0014, Rev. 6
Effective Date: 02/16/07
Cover Page

Title: Glassware Washing

[Method: None]

Approvals (Signature/Date):

Olga Colon	12/14/07		12-14-07
Technology Specialist	Date	Health & Safety Coordinator	Date
Dorothy J. Leson	12/13/07		12/14/07
Quality Assurance Manager	Date	Laboratory Director	Date

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STL North Canton
STANDARD OPERATION PROCEDURE

TITLE: GLASSWARE WASHING

(Supersedes: Revision 5, Dated 12/08/04)

Reviewed by:	Technology Specialist	5/9/07 Date
Approved by:	Quality Assurance Manager	5/22/07 Date
Approved by:	Environmental Health and Safety Coordinator	2-27-07 Date
Approved by:	Laboratory Director	5/9/07 Date

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1. PURPOSE

1.1. The procedure listed in this document will describe the standard operating procedures for washing glassware in the laboratories.

1.2. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. RESPONSIBILITIES

2.1. It is the responsibility of the analyst to perform the analysis described herein in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the analysis described is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the analyst to perform this SOP correctly.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

3.2. 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	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Material (1)	Hazards	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
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.
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.			

3.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves **MUST** be worn when washing glassware. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

3.4. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. Solvent and waste containers will be kept closed unless transfers are being made.

3.5. The glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported immediately to the EH&S Coordinator and to a Laboratory Supervisor.

3.7. Glassware in contact with chemicals used in analytical procedures may be toxic or carcinogenic. Therefore, each piece of glassware should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level.

3.8. Hands must not be placed in the glassware while washing. An appropriate scrub brush must be used to clean the inside of glassware. This will prevent the breakage of glassware while trying to force hands in or out of apparatus.

4. PROCEDURES

4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

4.2. Metals

4.2.1. Dirty glassware is taken to a central location and thoroughly rinsed. Any ink on the outside of the glassware is removed with acetone.

4.2.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware.

4.2.3. Rinse the glassware thoroughly three times with hot tap water and then rinse once with 1:1 nitric acid. Appropriate protective wear should be worn. If the glassware is still visibly dirty, or if spotting or beading occurs, repeat Section 4.2.2.

4.2.4. Rinse the glassware three times with analyte-free water, and place it in a clean drying area.

4.2.5. After air drying, the glassware should be spot- and stain-free. If not, then repeat the entire procedure. Seldom used items should be stored to minimize contamination.

4.3. Wet Chemistry

4.3.1. After using a piece of glassware, it is thoroughly rinsed with hot tap water and carried to the dirty glassware area. If visibly dirty, it should be washed immediately.

4.3.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware. A mechanical, laboratory dishwasher equipped with a DI water rinse is also approved for glassware cleaning.

4.3.3. Rinse the glassware thoroughly three times with hot tap water. If the glassware is still visibly dirty, or if spotting or beading occurs, repeat Section 4.3.2.

4.3.4. After air drying, the glassware should be spot- and stain-free. If not, then repeat the entire procedure.

4.3.5. Place the glassware in a designated clean storage location free from interferences or contamination.

4.4. Semivolatile Organics

4.4.1. If high level contamination is suspected perform the appropriate high level cleaning before proceeding with the standard cleaning process described in Section 4.4.2 and following.

4.4.1.1. Non-polar organics: if the glassware was most recently *wet* with a solvent, then rinse three times with that solvent, if *wet* with water, then rinse three times with acetone. Collect rinses in the appropriate solvent waste container. Allow the remaining solvent to evaporate from the glassware in a ventilation hood to reduce analyst exposure to the solvent.

4.4.2. After using a piece of glassware, it is thoroughly rinsed with hot tap water and carried to the dirty glassware area. If it is visibly dirty, it should be washed or soaked immediately rather than allow the residue to harden and become more difficult to wash.

4.4.3. The glassware is immersed in a hot, soapy solution of water and laboratory detergent (recommended pH > 10). An appropriate scrub brush is used to scrub the glassware. Change the wash water (when it becomes cold, visibly dirty or is used on glassware with oil or sediment residue) by emptying, rinsing the inside of the sink with hot water and refill with hot water and detergent.

4.4.4. Rinse the glassware three times vigorously with hot tap water. If the glassware is still visibly dirty, or if spotting or beading occurs, rinse with 1:1 HCl and repeat Section 4.4.3.

4.4.5. After completing the hot tap water rinse, rinse the glassware three times with deionized water and place it in a clean drying area.

4.4.6. Place cleaned glassware used for semivolatiles in a muffle oven for approximately one hour at 400°C. Do not heat glassware with visible residue. Baked-on residue is much more difficult to remove. Do not heat volumetric glassware, including flasks, pipettes, syringes, etc., to avoid deformation.

4.4.7. Clean glassware is inverted (where applicable) and stored prior to use in a designated clean storage location.

4.4.8. Before use, glassware is to be pre-rinsed with the solvent inherent to a given procedure if stored more than one week.

4.5. Volatile Organics

4.5.1. Rinse the glassware vigorously with hot tap water. If necessary, use a scrub brush to remove any remaining residue.

4.5.2. Place the cleaned glassware in the drying oven until dry or overnight.

4.5.3. Allow the cleaned and dry glassware to cool completely before use.

4.6. All Laboratory Groups

4.6.1. All glassware washing brushes must be hung on hooks when not in use to prevent contamination and ensure proper drying.

5. DEFINITIONS

5.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

6. POLLUTION PREVENTION

6.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

7. WASTE MANAGEMENT

7.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

7.2. Waste Streams Produced by the Method

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

7.2.1.1. Flammable Solvent Rinse. Used solvents generated from glassware cleaning operations are placed in waste containers identified as "Mixed Flammable Solvent Waste"

7.2.1.2. Spent Acid Rinse. Used acids generated from cleaning glassware are collected in containers identified as "Acid Waste".

8. APPENDICES

8.1. See Table 1, Glassware Washing

Table 1 Glassware Washing

	Metals	Organics	Wet Chemistry
Wash	Hot water, detergent solution	Hot water, detergent solution	Hot water, detergent solution
Rinse	3 times tap water 1 time 1:1 Nitric acid 3 times reagent water	3 times tap water 1 time 1:1 Hydrochloric acid* 3 times reagent water	3 times tap water
Dry	Air	Muffle at 400°C for one hour	Air
Storage	Designated cabinets and shelves	Designated cabinets and shelves	Designated cabinets and shelves

*If the glassware is visibly dirty after washing in a hot detergent solution, the glassware is rinsed with 1:1 HCl and rewashed with a hot detergent solution.

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Revision No. 3
Revision Date: 09/19/07

TESTAMERICA STANDARD OPERATING PROCEDURE

TITLE: NONCONFORMANCE AND CORRECTIVE ACTION SYSTEM

(SUPERSEDES: REVISION 2, DATED 06/15/99)

Approved by: Dorothy J. Leeson — 9/21/07
Quality Assurance Manager — Date

Approved by: [signature] — 9/21/07
Laboratory Director — Date

Approved by: Mark Bruce — 9/24/07
Technical Director — Date

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1. PURPOSE

1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The TestAmericaQuality requires documentation of instances of deviations from established control limits, approved standard operating procedure (SOPs), or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from TestAmerica policies and procedures and documented client specifications including root causes and corrective actions taken to remedy the nonconformance. The NCM may be stored in electronic form in a database, or maintained using defined printed forms.

1.2. This document applies to procedures, services, analytical data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. Nonconformances related to sample receiving activities may be documented separately from the NCM process by use of the Cooler Receipt Form (CRF) as described in a facility-specific SOP. Regardless of the type of system is used to handle the sample receiving and client-related issues, including holding time violations (HTVs), the system must include and emphasize the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.

1.3. Nonconformances can be identified by TestAmerica laboratory employees in the course of their daily operations or by external parties (i.e., customers and representatives of customers) through reviews of records, audit, or proficiency testing.

2. RESPONSIBILITIES

2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, that might affect the quality of TestAmerica's product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.

2.2. **Group Leader/Team Leader/Supervisor** (for purposes of this SOP, the term "Supervisor" will be used): Each Supervisor is responsible for the review of NCMs to ensure that problems which might affect quality are adequately described and that personnel are assigned to correct them. Supervisors review hardcopy or electronic versions of NCMs and forward them to the appropriate Project Manager. Together with Project Managers

and Quality Assurance personnel, Supervisors are responsible for determining the appropriateness of planned corrective actions.

2.3. **Project Manager (PM):** The Project Manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The Project Manager communicates conformance problems to clients and documents decisions made with clients. The Project Manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that repreparation and analysis of a sample was done. The Project Manager can and must withhold final reports to clients until corrective actions agreed to with the client have been completed.

2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for reviewing all initiated NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and spot audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.

2.5. **Operations/Systems Manager**: The operations/systems manager shall ensure that corrective actions are correct and have been implemented. The operations/systems manager shall document this review and concurrence by signing the NCM as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations/systems manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.

2.6. **Laboratory Manager:** The laboratory manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.

2.7. **Corporate QA Director**: The TestAmerica Quality Assurance Director should be notified of any continuing nonconformances that are not properly addressed by operations or where the root cause cannot be identified.

3. SAFETY

3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2. Procedures shall be carried out in a manner that protects the health and safety of all TestAmerica associates.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory Supervisor.

4. PROCEDURE

4.1. When to Initiate a Nonconformance Memo

4.1.1. Lab associates are to initiate an electronic nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or standard materials deviate from established specifications. All nonconformances with the exception of matrix-related failures require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions).

4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, bench sheets, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPPs Quality Assurance Summaries, or equivalent systems). Any intentional deviation from an SOP must be pre-approved by the QA manager and Supervisor. Any deviation from a SOP or client requirement not previously approved must be documented using the NCM process.

4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple lot numbers and samples, but normally a separate NCM would be initiated for different nonconformance issues. If the nonconformance involves projects for multiple Project Managers, then the NCM will need to be routed to each Project Manager.

4.1.4. Laboratories QuantIMS shall use the NCM database application developed for this purpose, **Clouseau**.

4.2. How to Process the NCM using Clouseau

4.2.1. Initiating the NCM

4.2.1.1. While properly logged in to a PC where Clouseau has been installed, start the Clouseau program.

4.2.1.2. At the Main Menu, select Create NCM.

4.2.1.3. Enter the information required at the top of the form. At a minimum select the Production Area, Classification (anomaly or deficiency), NCM Type, and NCM Description.

4.2.1.4. For NCMs that affected samples, indicate the relevant batch numbers, lots, samples, and tests by using the Tests/Samples option within Clouseau.

4.2.1.5. Complete the Comments/Details and Corrective Action sections of the Create NCM Form. These sections may be filled out jointly with the Supervisor. Consult the Project Manager or the operations/systems manager and QA Manager if the Supervisor and associate are uncertain of corrective actions. Be objective and specific. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information). Include enough information that details can be inserted directly into the Case Narrative section of the client report.

4.2.1.5.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples.

4.2.1.5.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.2.1.6. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff before the NCM is considered closed.

4.2.1.7. Save the NCM using the appropriate command in Clouseau. When presented with the Send E-Mail form, the Supervisor of the affected Production Area, the Project Manager, and the QA Department will be listed in the "Notify Now" box. Verify that these names are correct. If any personnel should be informed that are not listed, add their names to the "Notify Now" box by double-clicking on their names in the Personnel box.

4.2.1.8. Initiate the NCM notification process by selecting SEND EMAIL.

4.2.2. Supervisory Review and Approval

4.2.2.1. The Supervisor will receive notification of the NCM via e-mail. The Supervisor must log in to a computer workstation where Clouseau has been installed, and run Clouseau.

4.2.2.2. Using the Review NCM form, select the NCM to be reviewed and click on OPEN.

4.2.2.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. If the corrective action has not been determined, the situation must be referred to the Project Manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager needs to be made aware of the issues, the QA staff will notify the operations/systems manager using the Under Review/Send Email options of Clouseau.

4.2.2.4. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired. Refer to Section 4.3.5.

4.2.2.5. The Supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the Supervisor. This is Operations' commitment to rectify the problem. This will be verified by the QA staff and/or the Operations Managers.

4.2.2.6. The Supervisor selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process. If the NCM is for a holding time violation, a Project Manager ***must*** be notified ***immediately***.

4.2.3. Project Manager Review, Client Notification, and Project Documentation

4.2.3.1. The Project Manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The Project Manager shall indicate using the Client Notification Form in Clouseau whether client notification is required or not.

4.2.3.2. Record the result of the client contact or leave blank if client contact was not required. Indicate the date the contact was made. This must be done whether client notification is completed or not.

4.2.3.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. Notify the Supervisor of any changes made to the corrective action plan. The Project Manager's review of the NCM serves as documentation that the PM has read and reviewed the NCM and is aware of the corrective action plan.

4.2.3.4. The Project Manager selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process.

4.2.3.5. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned in to the QA staff if corrections are needed in the database (QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.2.3.6. After the QA staff has closed the NCM, the NCM is stored in the Clouseau program for future reference

4.2.4. Quality Assurance Review and Trending

4.2.4.1. The QA staff shall review all NCMs for conformance with standard laboratory practices.

4.2.4.2. NCMs will be reviewed to ensure that the corrective action was completed.

4.2.4.3. The Clouseau reporting and tracking system will be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.2.4.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic problems must be documented by the responsible laboratory area, along with supporting evidence.

4.2.4.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful.

4.2.4.6. The approval of the QA manager or designee is required in Clouseau to indicate that the NCM has been closed.

4.2.4.7. Clouseau will be the official database of all NCMs whether closed or pending for review and closure.

4.2.5. Instrument/Equipment Nonconformance Tag

4.2.5.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a clearly visible tag or sign indicating the nonconforming condition. (See Example in Attachment B.)

4.2.5.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM (electronic or hard copy). Identify the instrument by name and/or identification number and briefly describe the problem.

4.2.5.3. Upon saving the NCM, the NCM number indicated by Clouseau shall be recorded in the instrument's maintenance log.

4.2.5.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record the back to control information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM

5. DEFINITIONS

5.1. ***Nonconformance:*** an unplanned deviation from an established protocol or plan. The deviation may be the result of TestAmerica actions--then termed a **deficiency**, or the result of events beyond the control of TestAmerica--then termed an **anomaly**.

A nonconformance exists when:

5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrate a **systematic** deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed to matrix effects must be documented, but an NCM is not required.

5.1.2. A procedure is not performed as described in the applicable SOP or QA Policy, ***except*** in cases where the procedure has been performed according to a client-specified document TestAmerica has agreed to follow (e.g., EPA SOWs and QAPPs).

5.1.3. A practice or procedure is not performed as described according to a client or project document that TestAmerica has agreed to follow.

5.1.4. Purchased materials or services are determined to be defective and their use would effect data quality.

5.1.5. Holding time violations occur regardless of what or whose actions caused them.

5.1.6. A formal NCM is not required for routine instrument maintenance and malfunctions, which can be documented in instrument maintenance logbooks.

5.2. ***Corrective action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.

5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.

5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Reference Documents

6.1.1. TestAmerica Laboratory Quality Manual (LQM), current revision.

6.1.2. Clouseau Program Documentation.

6.2. Appendices

6.2.1. Attachment A: Instrument/Equipment Nonconformance Tag Form

ATTACHMENT A

EXAMPLE

INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG FORM

TESTAMERICA

CAUTION

DO NOT USE

NONCONFORMING ITEM

NCM NUMBER_____

AFFECTED ITEM _____

_____

_____

_____ _____
ANALYST DATE

WORK MAY NOT PROCEED ON THIS ITEM UNTIL SUCCESSFUL CALIBRATION IS DOCUMENTED.

Controlled Copy No. _____

Implementation Date: 9-24-07

Policy No.: QA-003
Revision No.: 6
Revision Date: 09/19/07
Page 1 of 20

APPROVAL:

Dorothy J. Leeson, Quality Assurance Manager

Opal Davis-Johnson, Laboratory Director

TESTAMERICA NORTH CANTON POLICY
Quality Control Program

Supersedes: Revision 5, Revision Date 09/27/04

OBJECTIVE:

This policy describes the TestAmerica North Canton program of routine analytical quality control (QC) activities. The objective is to generate QC data that demonstrate that the analytical process is in control and that the data meet client and method requirements. The policy outlines QC requirements for a variety of regulatory programs, with the stipulation that when lacking specific direction from our clients, TestAmerica North Canton will default to routine RCRA program QC requirements.

SCOPE:

This policy is to be enforced and followed throughout the laboratory.

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POLICY:

1. Assessments of QC data relative to control limits determine the acceptability of sample test results. Whenever control criteria are not met, the data must be evaluated to determine appropriate corrective action. The initial evaluation is made by the analyst, frequently in conjunction with data review software and/or senior analysts or supervisors. Further technical evaluation of the data or data review software output is conducted by second-party data reviewers. Corrective action decisions, particularly whether or not to reanalyze samples, should be done in consultation with the client to the extent possible when operating under project-specific QA plans. Requirements for assessment and corrective action are described in the attachments to this policy. Details concerning technical data review and documentation of the reviews are described in the Laboratory Quality manual (LQM).

2. The TestAmerica North Canton standard QC program is to be communicated to the client prior to acceptance of work. At the same time, every effort must be made to understand the client's special project requirements. Generally, laboratory project managers serve as a liaison between the clients and the laboratory staff to ensure that requirements are properly communicated in writing to both parties. In the event that alternative QC procedures are not specified by our clients, these standard QC protocols must be followed to ensure the generation of legally and scientifically defensible analytical data.

3. Successful implementation of this QC program requires that it is clearly understood by all TestAmerica staff. Training based on this policy will be conducted periodically and provided to new personnel as appropriate for their functions.

4. TestAmerica North Canton QC program applies to the following:

   RCRA and SW-846 Projects

   All routine analytical projects performed using SW-846 methods must comply with the requirements described in the TestAmerica North Canton Laboratory Quality Manual (LQM) and Attachment I to this policy. The Quality Control sections of analytical standard operating procedures (SOPs) referencing SW-846 methods must be consistent with the requirements in Attachment I.

   CWA and 40 CFR Part 136 Projects

   Any analytical work conducted in support of an NPDES permit or other Clean Water Act compliance activities, must meet the quality control specifications shown in the LQM. The quality control requirements for the specific methods listed in the LQM define the minimum requirements that must be given in laboratory analytical SOPs.

   Other Programs or Projects with Clearly Defined QC Requirements

   The differences between the TestAmerica North Canton standard QC program and special project requirements must be specified in project documents. These documents may include Quality Assurance Project Plans (QAPjPs), Quality Assurance Program Plans

(QAPPs), Sampling and Analysis Plans (SAPs), project-specific Quality Assurance Summaries (QASs), SOPs, contracts, or other approved documents.

Documents describing special project requirements must be reviewed and approved by appropriate QA and operations staff.

If the special project requirements appear to result in modifications that contradict federal or state regulatory requirements, the variance must be noted in writing and communicated to the client. A record of this communication must be retained as a permanent part of the project file.

Any special client project requirements must be communicated to TestAmerica North Canton's analysts in advance of releasing samples for analysis, and the work must be clearly differentiated in the analytical documentation, otherwise Attachment I requirements will be followed.

Projects Without Specific QC Requirements

Any projects for which no specific QC program is specified must follow the requirements shown in Attachment I.

5. Analytical SOPs must include a quality control section that addresses these general QC requirements. As relevant, specific method QC requirements should be given precedence to these general requirements.

ATTACHMENT I

QC for RCRA PROJECTS AND PROJECTS WITHOUT DEFINED QC REQUIREMENTS

1.0 Introduction

This Quality Control (QC) Program is based on the requirements in "Test Methods for Evaluating Solid Waste", USEPA SW-846, Third Edition with promulgated updates. It applies whenever SW-846 analytical methods are used. It also applies in whole or in part whenever project requirements fail to specify some aspect of QC practices described here. It does not apply when other well defined QC programs (e.g., DoD QSM) are specified. This policy represents TestAmerica North Canton base QC program for environmental analyses.

Details concerning instrument calibrations, tunes, and QC that are required for specific methods (e.g., interference check samples for ICP) are not given here. Refer to the method standard operating procedures (SOPs) for information about the frequency, assessment and corrective action required for additional QC elements.

2.0 Definitions

2.1 Batch Definition – a batch is a group of no greater than 20 samples, excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents, the same processes, and the same personnel.

2.2 Surrogates - Surrogates are organic compounds similar in chemical behavior to the target analytes, but that are not normally found in environmental samples. Surrogates are added to all samples in a batch to monitor the effects of both the matrix and the analytical process on accuracy.

2.3 Method Blank - The method blank (MB) is a control sample prepared using the same reagents used for the samples. As part of a QC batch, it accompanies the samples through all steps of the analytical procedure. The method blank is used to monitor the level of contamination introduced to a batch of samples as a result of laboratory processing.

2.4 Instrument/Calibration Blank - The instrument blank is prepared using the same solvents and reagents (e.g. hexane, methylene chloride, or reagent water) used to dilute the prepared sample extracts or digests. Unlike the method blank, it is analyzed without being subject to the

preparation steps of the analytical procedure. It is used to monitor laboratory or reagent contamination introduced at the instrumental analysis phase of work. For procedures without a separate preparation step, an instrument blank is equivalent to the method blank, and serves the same purpose.

2.5 Laboratory Control Sample - A laboratory control sample (LCS) is prepared using a well characterized matrix (e.g. reagent water or Ottawa sand) that is spiked, with known amounts of representative analytes. Alternate matrices (e.g. glass beads) may be used for soil analyses when Ottawa sand is not appropriate. As part of a QC batch, it accompanies the samples through all steps of the analytical process. The LCS is used to monitor the accuracy of the analytical process independent of possible interference effects due to sample matrix. Information regarding precision of the method can be determined over time.

2.6 Matrix Spike and Matrix Spike Duplicate

2.6.1 Matrix Spike - A matrix spike (MS) is a replicate portion of one field sample in the QC batch that is spiked with known amounts of target analytes. An MS is spiked with the same analytes at the same concentrations that are added to the LCS. Any client sample that is not a field blank can be used for a matrix spike as long as there is sufficient quantity. As part of the QC batch, it accompanies the field samples through all steps of the analytical process. Matrix spike data are only meaningful for the sample in which they are prepared and samples from the same site.

2.6.2 Matrix Spike Duplicate - A matrix spike duplicate (MSD) consists of an additional portion of the same sample used to prepare the MS. This portion is spiked and processed exactly as the MS.

2.6.3 The MS and MSD results are used to determine the effect of the sample matrix on the precision and accuracy of results. Due to the potential variability of the matrix of each sample, the MS and MSD results may not have immediate bearing on any samples except the one spiked.

2.7 Sample Duplicate - A sample duplicate is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. Any client sample that is not a field blank can be used for a sample duplicate as long as there is sufficient quantity. The sample and duplicate results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample duplicate precision results are not necessarily representative of the precision for other samples in the batch.

2.8 Duplicate Control Sample - A duplicate laboratory control sample (LCSD or DCS) may be prepared at the request of the client. It is required for some projects particularly when insufficient sample volume is received to prepare and analyze an MS/MSD pair. LCS/LCSD pairs provide additional information regarding the precision of the measurement process.

3.0 Batch QC Elements & Batch Processing

3.1 A QC batch is designed to determine the quality of the analytical results obtained for a group of up to 20 field samples in terms of accuracy and precision. With some exceptions as described in Sections 3.6 through 3.8 below, the minimum QC elements for each QC batch are

- one method blank (MB),
- one laboratory control sample (LCS),
- one matrix spike (MS), and
- one matrix spike duplicate (MSD).

3.2 The identity of each QC batch must be documented and traceable, i.e., each batch of field samples must be clearly associated with the applicable QC samples.

3.3 To the extent possible, samples that require a preparation step should be analyzed together with their associated QC samples. If the samples in a given QC batch require separate analytical runs, the minimum batch QC in each run is an acceptable MB or instrument/calibration blank.

3.4 For analytical procedures that do not include a separate extraction or digestion (e.g., volatile organic analysis by purge and trap), the QC batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

3.5 Field QC samples (e.g., trip blanks, equipment rinsates, and field duplicates) count as individual samples, therefore, they add to the QC batch count. Samples that require simple reanalysis (e.g., dilutions to adjust a sample extract to the working range of the instrument), as opposed to reextraction or digestion and reanalysis, do not count as additional samples in the QC batch. For procedures without a separate preparation, a reanalysis within the same calibration event (as defined in Section 3.4) does not add to the batch count.

3.6 MS/MSD pairs are not the only acceptable means of demonstrating precision.

3.6.1 As requested by clients or required by some methods, batch precision may also be demonstrated through the analysis of sample duplicates. However, the client should be

advised that a duplicate is less likely to provide usable precision statistics depending on the likelihood of finding concentrations below reporting limits.

3.6.2 A duplicate LCS (LCSD or DCS) may be used to demonstrate method batch precision independent of client's matrix. LCSDs are prepared at the client's request, and are often used when the client has not supplied sufficient sample quantity to prepare an MS, MSD or duplicate.

3.6.3 On-going monitoring of LCS results can be used to determine long-term precision and accuracy for a method.

3.7 Some methods including isotope-dilution methods, pH and ignitability for example, do not use all of the QC elements listed in Section 3.1. Method exceptions to these requirements are listed in the TestAmerica North Canton LQM QC tables and in the laboratory analytical SOPs.

3.8 Deviations from these QC elements must either be noted in project planning documents (QAPPs, QAPjPs, SAPs, SOWs, QAS, or equivalent) or in a nonconformance memo (see SOP CORP-QA-0010 for details).

4.0 Data Evaluation and Corrective Action

4.1 General Guidelines

4.1.1 Any QC component that is outside of established control limits is considered an out-of-control event. All out-of-control events must be documented and the associated data evaluated. Depending on the specific circumstances, evaluation can lead to a variety of actions. The following sections and the flowcharts describe the appropriate corrective action for the most common QC failures. However, it is not possible to address all possible data evaluation scenarios in this policy. The guiding principle for all evaluations is that the data and corrective action decisions must be defensible using TestAmerica North Canton policies, procedures or scientific evidence, and justified in the project records.

4.1.2 If reanalysis for QC failures is conducted and the second analysis confirms a QC problem that is outside of the laboratory's control, further testing is not necessary. The problem must be documented and the data properly qualified in the project report.

4.1.3 QC failures that are not corrected by reanalysis are documented in the TestAmerica North Canton electronic Nonconformance System (Clouseau) as described in SOP CORP-QA-0010.

4.1.4 QC failures due to sample matrix interferences (particularly MS, MSD and sample surrogate failures) do not have to be documented in the Clouseau system unless there is a holding time violation. Other forms (e.g., Organic Data Review Checklist) may be used to document matrix QC failures. In either case, matrix QC failures must be communicated to the laboratory project manager and significant matrix QC failures must be discussed in the final report case narrative.

4.1.4 When ongoing, systematic problems are identified, work must stop until it can be demonstrated that the system is in control again.

4.2 Method Blank (MB) Evaluation (see Figure 1)

4.2.1 Method Blank Acceptance Criteria

The results of the method blank shall be one of the QC measures used to assess batch acceptance. Results are acceptable if all analyte concentrations in the MB meet the following criteria:

- Organics -The blank contamination is less than 1/10 of the measured concentration of any sample in the associated preparation batch , or
- Inorganics – The blank contamination is less than 1/20 of the measured concentration of any sample in the associated preparation batch, or
- The blank contamination is less than the concentration present in the samples and is less than 1/10 of the regulatory limit, or
- The same contaminants were not found in the associated samples, or
- MB results are less than or equal to the reporting limit.

Note: Positive method blank results slightly below the reporting limit should still be evaluated by the analyst for potential impact on sample results at or near the reporting limit.

Note: DoD project requirements are noted in NC-QA-0016, Supplemental Practices for DoD Project Work.

Note: For Ohio VAP projects, the method blank contamination must be below the reporting limit.

4.2.2 Corrective Action for Method Blank Failure

- If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. Samples associated with the contaminated blank shall be reprocessed for analysis or, under the following circumstances, may be reported as qualified (qualifier flags or narrative comments):

- MB contamination at a level less than the reporting limit with sample results at levels near the RL, based on analyst's judgement shall be flagged, if flags are requested by client.
- Analyte concentrations in samples are greater than 10 times blank contamination for Organics and 20 times the blank contamination for Inorganics, or
- The contaminant is a common blank contaminant (see below) and the MB concentration is less than 5 times the RL for organics or less than two times the RL for inorganics. Note that some programs do not recognize common lab contaminants.

Common Laboratory Contaminants:

Analyte	Method
Methylene Chloride	Volatile Organics (GC or GC/MS)
Acetone	Volatile Organics (GC or GC/MS)
2-Butanone	Volatile Organics (GC or GC/MS)
Phthalate Esters	Semi-Volatile Organics (GC or GC/MS)
Copper	Metals (ICP or ICPMS)
Zinc	Metals (ICP or ICPMS)
Iron	Metals (ICP or ICPMS)
Lead	Metals (Trace ICP or ICPMS)
Barium	Metals (ICPMS)
Chromium	Metals (ICPMS)
Manganese	Metals (ICPMS)
Calcium	Metals (ICPMS)
Magnesium	Metals (ICPMS)
Potassium	Metals (ICPMS)
Sodium	Metals (ICPMS)

4.3 Laboratory Control Samples (LCS) Evaluation (see Figure 2)

4.3.1 Acceptance Criteria

The LCS recovery for the control analytes must be within established control limits. The percent recovery is calculated as follows:

$$\text{LCS Percent Recovery} = \frac{X}{t} \times 100$$

Where: X = observed concentration
t = concentration of spike added

4.3.2 Corrective Action for LCS Failure

- check calculations,
- check instrument performance,
- reanalyze the LCS, and if still outside of control limits,
- reprepare and reanalyze all samples in the QC batch.

Notes: 1. It is acceptable to report the data if the LCS recovery is out high and analyte of concern was not detected in any of the samples.

2. In the case of volatile analyses, if the LCS fails, a new LCS may be reprepared and reanalyzed within the same tune period.

3. In the case where all target requested analytes are within control, but some other LCS compounds are out of control, the LCS may still be considered acceptable for reporting.

4.4 Duplicate Laboratory Control Samples (LCS/LCSD or DCS) Evaluation (see Fig. 2)

4.4.1 Acceptance Criteria

The recovery for each spike of the pair must be within established control limits. The formula used to calculate LCSD recoveries is the same as the formula for LCS spike recoveries. If a batch includes samples requiring LCS control and samples requiring both LCSs and LCSDs, the LCS used will be the first LCS that passes control criteria.

The relative percent difference (RPD) for the pair is calculated as follows:

$$\text{RPD} = \left[\frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} \right] \times 100$$

Where: X_1 = first observed concentration

X_2 = second observed concentration

4.4.2 Corrective Action for LCS/LCSD Recovery (Accuracy) Failure

- check calculations,
- check instrument performance,
- reanalyze and/or reprepare and reanalyze all samples in the QC batch.

Note: If either LCS/LCSD spikes fails and the batch cannot be reanalyzed, the failure must be documented and noted in the final report. Also see notes under Section 4.3.2.

4.4.3 Corrective Action for LCSD Precision Failure

- Check calculations
- Check instrument performance
- If the RPD is out of control, but both accuracy recoveries are within acceptance criteria, prepare an NCM and qualify report.

Note: Because LCS/LCSD limits are based on the standard deviation of data collected over time and include long-term precision, it would be unusual to fail precision limits while meeting accuracy limits. If this occurs with any frequency, control limits should be reevaluated.

4.5 Surrogate Evaluation (see Figure 3)

4.5.1 Acceptance Criteria

Surrogate recoveries must be within established control limits. Method QC (MB, LCS, and/or LCSD) results are not acceptable unless the surrogate recoveries for those QC samples are within control limits. If MS/MSD, duplicate or field samples require dilutions beyond the threshold stated in the analytical SOPs, routine surrogate control limits do not apply and recoveries are not evaluated. This should be noted in the final report. The recovery is calculated as follows:

$$\text{Surrogate Percent Recovery} = \frac{X}{t} \times 100$$

Where: X = observed concentration
t = concentration of surrogate added

4.5.2 Corrective Action

4.5.2.1 Surrogate Failures in MB, LCS, or LCSD

- check calculation and instrument performance,
- reanalyze QC sample and/or reanalyze all samples in the QC batch.

Note: For Ohio VAP projects, the batch must be re-extracted if reanalysis does not resolve the problem.

4.5.2.2 Surrogate Failures in Samples or MS/MSD

- check calculation and instrument performance
- evaluate objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses)
- document the failure and note it on the final report

Note: Unless otherwise specified by the client, it may be possible to report qualified results if method QC surrogate recoveries are biased high and analytes were not detected in the field samples. However, all other QC requirements would have to be met and the failure would have to be noted in the final report.

Note: Some client programs require reanalysis to confirm matrix interferences. Check special project instructions for this corrective action.

4.6 Matrix Spike and Matrix Spike Duplicates (MS/MSD) Evaluation (see Figure 4)

4.6.1 Acceptance Criteria

MS and MSD recoveries and RPD should be within established control limits. If MS or MSD samples require dilutions beyond the threshold stated in the analytical SOPs, routine control limits do not apply and recoveries are not evaluated, but this should be noted in the final report. The MS and MSD recoveries are calculated as follows:

$$\text{MS or MSD Percent Recovery} = \left[\frac{X_s - X}{t}\right] \times 100$$

Where: X = observed concentration in unspiked sample
X_s = observed concentration in spiked sample
t = concentration of spike added

Note: 1. If sample result is ND, $X = 0$ when no values reported below RL. If sample result is reported as a value <RL, X = reported value.

2. CLP forms software uses observed recovery, not concentrations.

RPD is defined in Section 4.4.1.

4.6.2 Corrective Action for MS/MSD or MS/MSD RPD Failure (assuming that the LCS is in control)

- check calculation and instrument performance,
- consider objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses);
- document the failure and note on final report;

Note: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

4.7 Sample Duplicate

4.7.1 Acceptance Criteria
The RPD for the sample and its duplicate must be within established control limits. The RPD is the same as for the MS/MSD (see Section 4.6.1).

4.7.2 Corrective Action for Duplicate Failure

- check calculation and instrument performance,
- document the QC failure and note on the final report.

5.0 Establishing QC Acceptance Limits

5.1 Initial Control Limits
For new procedures, published method limits can be used until sufficient QC data are acquired (minimum of 20 to 30 data points recommended). However, the published limits may not be appropriate if they are based on a single-operator or single-laboratory study. In this case, the QA Manager may establish default limits until enough data is collected for laboratory established limits to be determined.

5.2 Control limits should be reexamined annually, and reset as needed. If the recalculated limits are consistent with the historical limits, the historical limits may remain unchanged.

5.2 Running the Control Limits Program

Evaluating control charts is an important first step in considering new control limits. This is done with the TraQAr Control Limits program. Only QA personnel who are familiar with the organization of TestAmerica North Canton spike lists are authorized to set control limits. The program collects a specified set of QC data, performs a Grubbs Outlier Test, calculates three standard deviation control limits, compares those limits to the existing limits in the laboratory LIMS, and generates an I-type control chart (ref. ASTM D 6299). This control chart is a plot of results in chronological order to which existing control limits and a centerline have been added. The control chart aids in the examination of the data to be sure that it is representative and appropriate for use in setting new limits. Refer to SOP NC-QA-0018, Statistical Evaluation of Data & Development of Control Charts for complete details, but some specific requirements include the following:

5.2.1 Select QC Type Options:

LCS/DCS - normally used to establish both LCS and MS/MSD control limits.

LCS/DCS Surrogates - normally used to establish surrogate control limits for both LCS and MS/MSD controls. It is rarely used.

MS/MSD - used to set matrix specific control limits, but use of such limits at TestAmerica Denver is generally restricted to materials from a particular site, and currently there is only one example for one client active in the system; this is used rarely.

MS/MSD Surrgates – as above, rarely used

All Surrogates – this option will produce a pooled set of LCS/LCSD, MB, MS/MSD, and sample surrogate results. It is the standard choice for creating surrogate control limits.

5.2.2 Grubbs Outlier Test

The Control Limits program automatically runs the test using a 5% level of significance, i.e., risk of falsely rejecting a data point. The test calculates a value for T based on the difference of the suspect point from the mean value, quantity divided by the calculated standard deviation.

$$T = \frac{|X_i - \overline{X}|}{s}$$

where, X_i is the point being considered for rejection

X bar is the mean, and

s is the standard deviation

The point is rejected if:

$$T > \frac{(N-1)}{\sqrt{N}} \sqrt{\frac{t^2_{(\alpha/2N),N-2)}}{N-2+t^2_{(\alpha/2N),N-2)}}}, \qquad \text{where N = number of points}$$

t = t distribution

Tables for critical values of T are given in John Taylor, Quality Assurance of Chemical Measurements, Lewis Publishers; 1987. If the measured value of T is greater than the value in the critical value, X_i is rejected. This assumes a normal distribution. (see www.itl.nist.gov/div898/handbook/eda for details about the derivation of the critical values of T).

5.3 Examine and Investigate Collected Data

Assuming that an adequate amount of data are collected, the next step involves determining that the data set is representative of the lab's performance, and therefore provides a useful prediction of future performance. A key part of the process is examining the data for bias, discontinuities, and/or trends. Ideally, if conditions are constant over the time period selected and existing limits are appropriate, the data will be evenly distributed around the centerline, with a few points at or slightly outside control limits. The reasons for deviations from the ideal should be investigated to be sure that the collected data are appropriate. Specific conditions requiring further investigation include data sets with no outliers, data with significant bias relative to existing limits, excessive number of outliers, discontinuous patterns, and upward or downward sloping trends.

5.4 Selecting New Control Limits

Generally control limits are based on the following statistics for the historical data

Accuracy: mean recovery $\pm 3s$
Precision: zero to (mean RPD + 3s)

Where: s = standard deviation

If the calculated 3 standard deviation limits are tighter than the method calibration verification criterion (e.g., CCV acceptance limits for ICP = $\pm$ 10% of expected value), then the new limits are set to the mean value $\pm$ calibration criterion.

5.5 Communicating and Implementing New Control Limits

QA personnel prepare control limit reports comparing the new control limits with the old. This information is sent to the supervisor of the area affected by the new limits. The supervisor is to

review the summary data and sign the documentation to confirm that the data selected are representative of current performance. The supervisor is also confirming that the instrument data systems will be updated on the implementation date--the same date that QuantIMS will be updated. The information is forwarded to clients who have requested it.

6.0 Reporting QC Data

QC data routinely reported with sample results include the LCS, method blank and surrogate standards. Client reporting format requirements are negotiable and documented as part of the project records. Ultimately, all reporting decisions should accommodate the client's requirements.

Figure 1 - Method Blank Evaluation

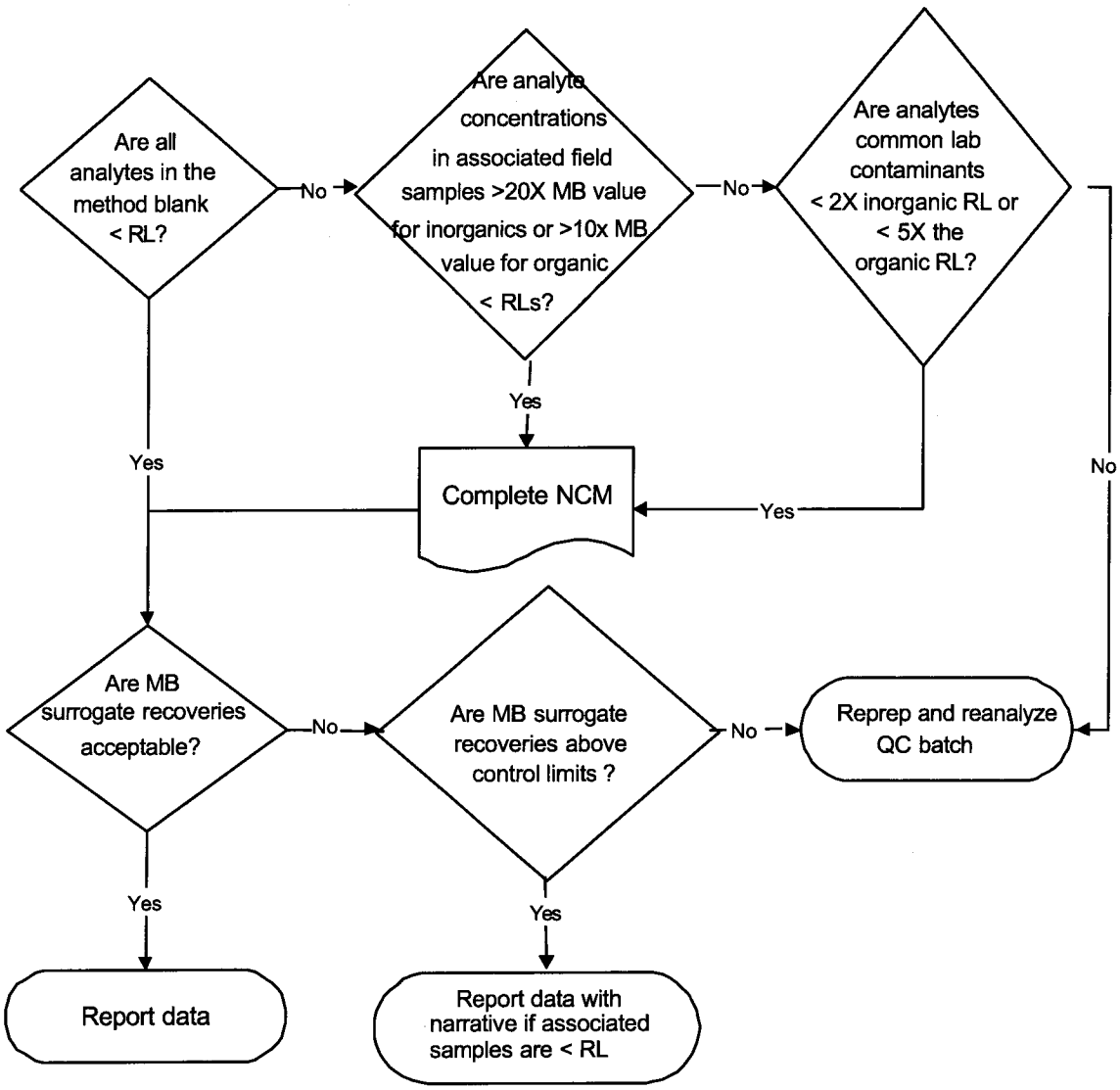


Figure 2 - LCS/LCSD Evaluation

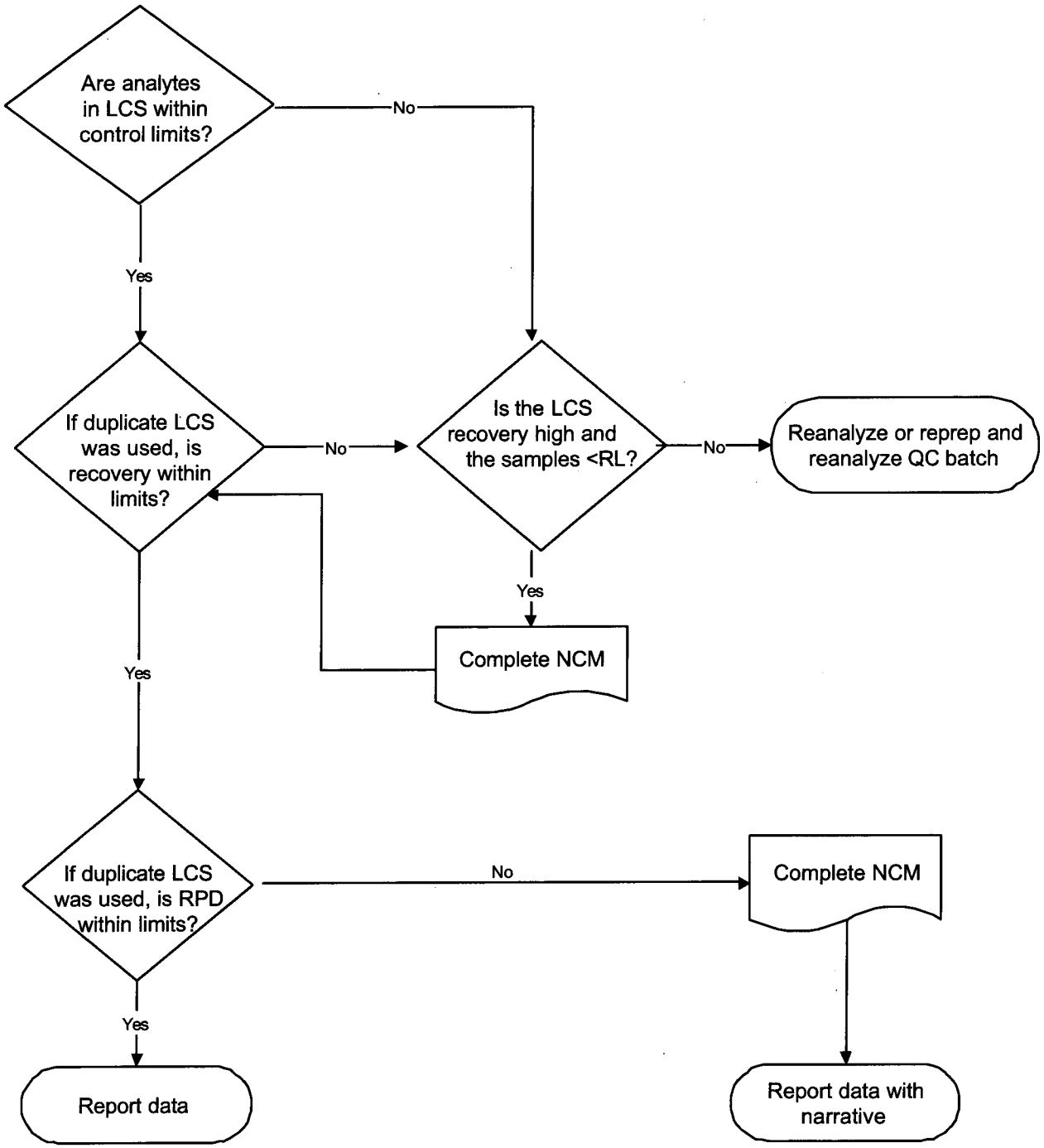


Figure 3 - Surrogate Evaluation

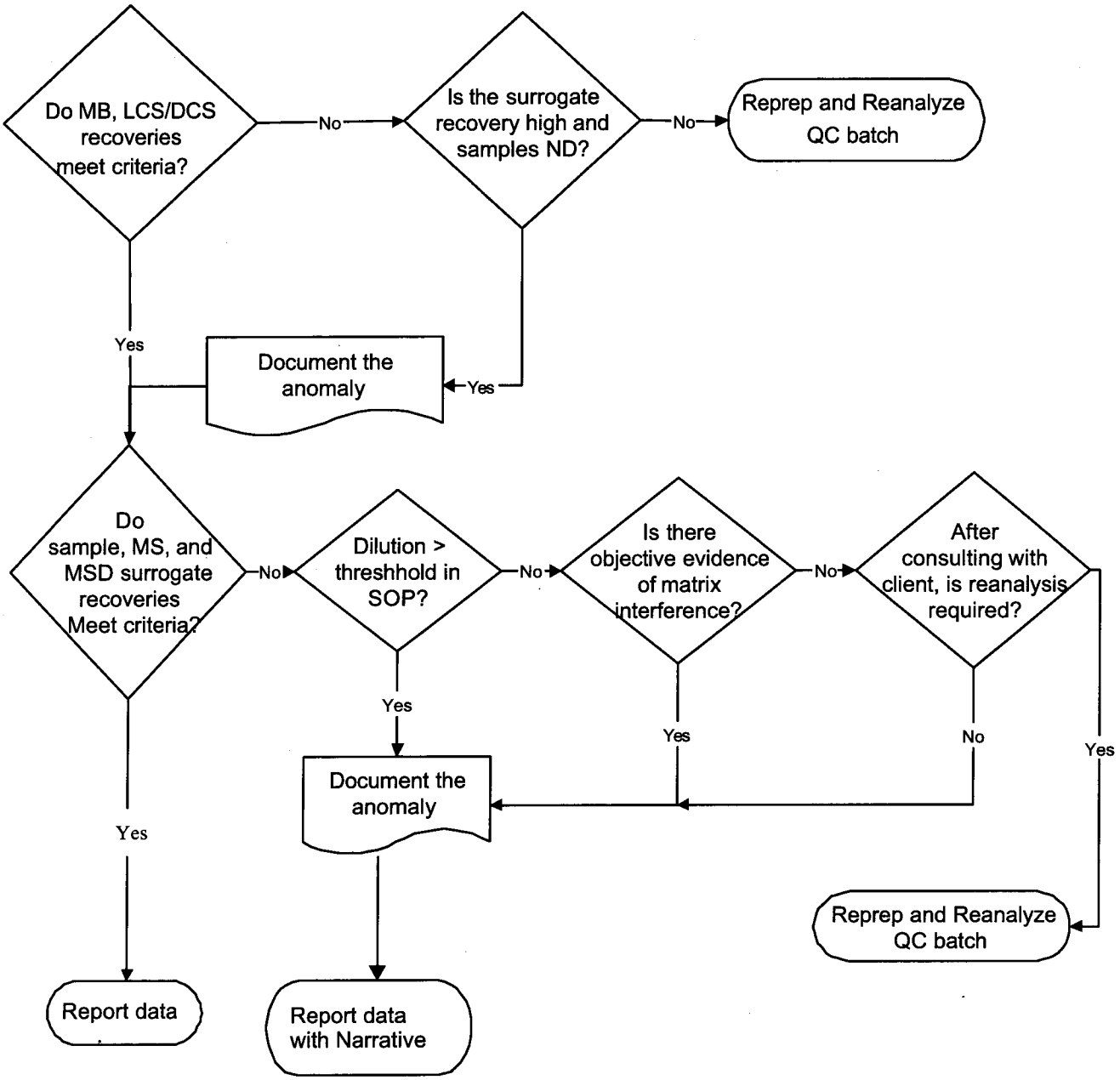
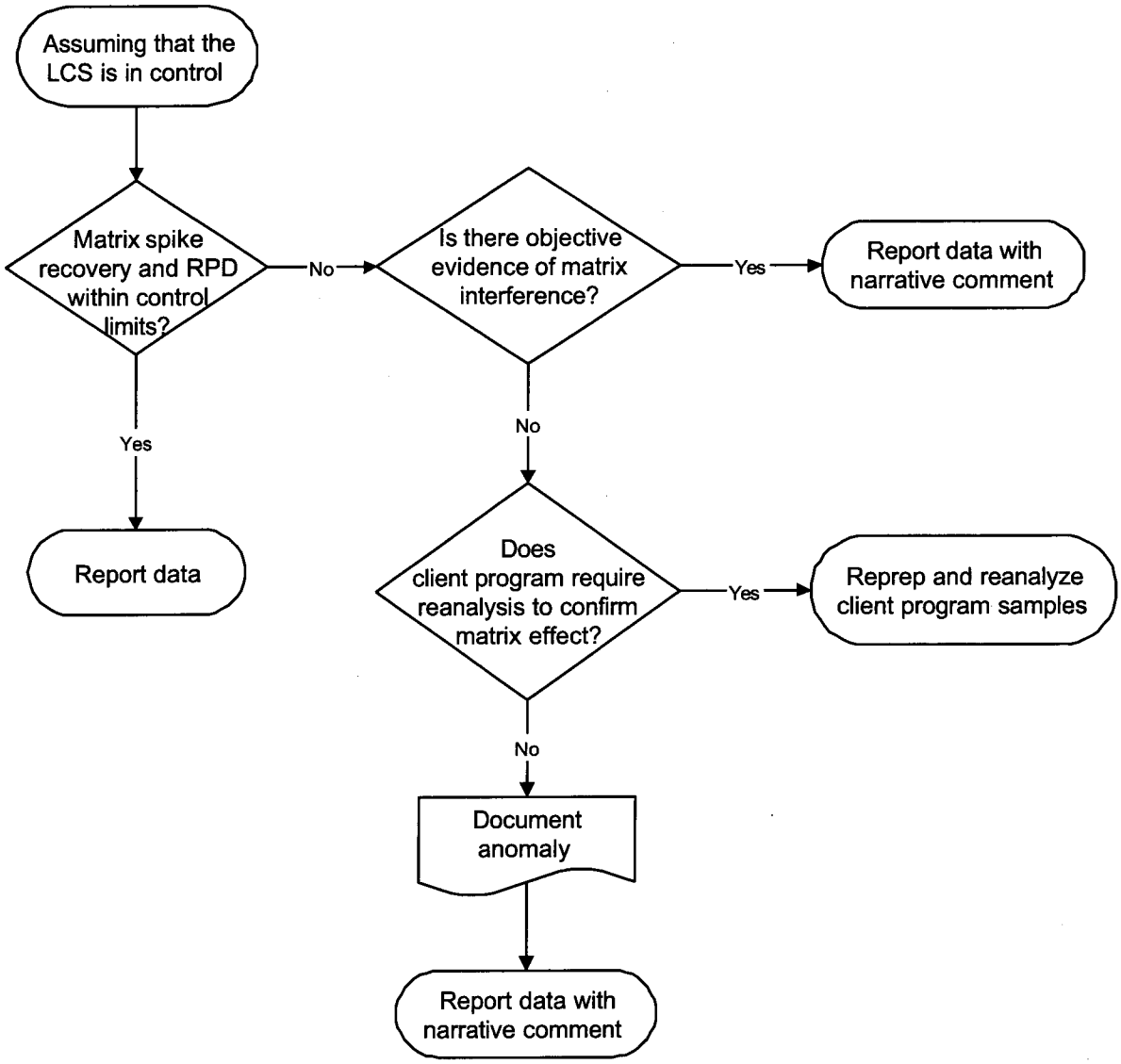


Figure 4 - Matrix Spike/Matrix Spike Duplicate Evaluation



TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

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Title: Records Information Management

[Method: None]

Approvals (Signature/Date):

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STL STANDARD OPERATING PROCEDURE

TITLE: RECORDS INFORMATION MANAGEMENT

(SUPERSEDES: REVISION 5, DATED 5/12/05)

Approved by: Lance Huhman — 2-27-07
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1. PURPOSE

1.1. This document outlines the procedures associated with the storage and maintenance of all record created in the normal course of business such as laboratory data, client project files and all records generated in the course of laboratory operations.

1.2. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. RESPONSIBILITIES

2.1. It is the responsibility of the employee to perform the procedure described herein in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of this facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

2.3. Records are stored in a secure area at all times and can only be removed if charged out.

3. SAFETY

3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2. Only 1.2 cubic foot boxes may be used to store all paper records due to risk of injury from larger sized boxes. These boxes have been found to weigh approximately 40 pounds on the average.

3.3. When the forklift is used, individuals will have forklift training under OSHA standards. All safety equipment, such as a safety harness, will be used when operating the forklift. Procedures

4. PROCEDURES

4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

4.2. New Lab ID Labels– to be printed out every day

4.2.1. At the Main Menu in QuantIMS, type "**FIL**" and press **enter**. At the command line, type "**F04**" (New Lab ID Label). The Lab ID (lot number) for the previous day has to be entered on both lines. If only one label is needed, the same lab ID is entered on both lines. For a range of labels, type the beginning range lab ID on the first line and the ending range lab ID on the second line; press "**Enter**." Labels are to be printed daily from samples received the previous day in Sample Receiving.

4.2.1.1. On the top line, enter:

4.2.1.1.1. Laboratory Location (**A** = North Canton)

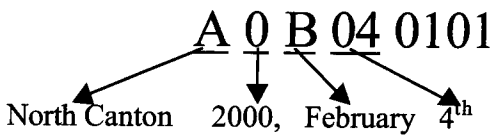
4.2.1.1.2. Last digit of the year (2000 = **0**, 2001 = **1**)

4.2.1.1.3. Month designation (**A** = January, **B** = February, etc.)

4.2.1.1.4. Previous day's date (5th = 05, 28th = 28)

4.2.1.1.5. "**0101**" (to designate the first sample of the day)

4.2.1.1.5.1. For example: A0B040101 means



4.2.1.2. On the bottom line, enter:

4.2.1.2.1. Repeat 4.2.1.1.1 - 4.2.1.1.4

4.2.1.2.2. Replace "**101**" with "**999**" (to designate the last sample of the day)

4.2.1.3. When completed, it will look like: A0B040101 – A0B04099

4.3. Labeling Folders and Filing

4.3.1. The receptionist will align the client labels at the top score mark on the side-tab manila folders.

4.3.2. Folders are then given centralized reporting to be filed and initiate report and chromatograms. All reports and chromatograms are filed together to form a "Project File".

4.4. Records Transfer Process

4.4.1. Record transfer forms are received in Record Management from all departments of the lab: Accounting, QA, EH&S, lab groups, etc. The reports/lab data is placed in a box by year, random order retention period and a record transfer form is created for each box.

4.4.2. The box is assigned an accession number (Cintas bar-coded label) and assigned a box location number. The date the reports are received are also written on the outside of the box, along with the client code range, Lab ID range and location. The label is stapled to the Record Transfer Form and both the accession number and box location is written on the form, copied and returned to the Record Liaison. Then at scheduled times the boxes that have been given labels are picked up a shelved.

4.4.3. At no time will the Records Manager check to see if the information listed on the record transfer form is correct. It is the responsibility of the record liaison to ensure the information is correct on the record transfer form.

4.5. Data Entry

4.5.1. Open up Report Tracker, click on "Menu", and click on "New Batch".

4.5.2. The next batch number will automatically appear.

4.5.3. In the Customer Number Box, click on the down arrow; and select the appropriate customer code as it appears on the Record Transfer form.

4.5.4. Entry date will automatically default to the current date.

4.5.5. Click on the arrow, and the next screen will appear for adding a new ascension number.

4.5.6. Click on "Add New Ascension"

4.5.7. In the Ascension Number field, enter the Andrews bar code number that was assigned to the Transfer form. Click "OK".

4.5.8. Click on the down arrow in the Record Series column. Select and click. Series will appear in the box.

4.5.9. Enter the box location that was assigned on the Record Transfer form. Example: 30-D-2A.

4.5.10. In the Document Description box, enter the information that is located on the Record Title of the Record Transfer for (no more than 50 characters).

4.5.11. In the "Date Range From" field, enter the earliest month as it appears on the Record Transfer form. Example: January would be entered as 01/01/05.

4.5.12. In the "Date Range To" field, enter the latest month and last day of that month as it appears on the Record Transfer form. Example: January would be entered as 01/31/05.

4.5.13. Click on the "Review Date" field, delete the current date, enter the last month and day as the "Date Range To" field, enter the year as it appears on "Destruction Date" on the Record Transfer form. Example: If the month "From and To" were January, and the Destruction Date was a five-year retention, in the Review Date field, it would appear as 01/31/05.

4.5.14. If there are no details to add in the "Detail" field, click the arrow to next field and click back again. This will save all data entry. If you need to add another ascension number, repeat steps starting from Section 4.5.5.

4.5.15. If there are details to enter (such as Client Code, Lab I.D., etc.), after entering in the "Add New Ascension" field is complete, click the arrow at the bottom of the field. This will take you to the Details field.

4.5.16. Enter: Client Code, Lab I.D., Title, Case Number, SDG, if applicable. As it appears on the Record Transfer form. Once this is complete, click on arrow back to the "Add New Ascension" field.

4.5.17. If another ascension number is to be added, return to Section 4.5.5 and repeat.

4.5.18. After all data entry is finished in that batch, click arrows left and select "Print Batch".

4.5.19. Print the batch out, and make two copies of each Record Transfer form. The original and one copy go to the originator of the form. The second copy and the Print Batch sheets are for the Records/Warehouse Manager's records. The Records Manager will Level I Review his copy with the Print Batch sheet, and make corrections as needed. He/she will then sign the "Print Batch" sheet as follows: "Level I Review, the date of review, and his/her initials". It will then be forwarded for Level II Review.

4.6. Editing a Batch

4.6.1. Open the Report Tracker. Click on "Edit Batch".

4.6.2. Enter the Batch Number. Click "OK". Click on the arrow, then click on the ascension number you wish to edit. Make the changes that are needed, and click arrow right to save changes. If changes need done in the "Detail" field, click arrow, make changes, and click arrows back left to save the changes.

4.7. Searching in the Report Tracker

4.7.1. Open Report Tracker. Click "Search". Enter details in the correct field, then click "Search".

4.7.2. Details can only be entered in one field at a time, except in the "Client Code" and "Lab I.D." fields. Multiple I.D. numbers and client codes can be entered here.

4.7.3. Once complete, in the "Search" mode, click "Back". This will return you back to the "Search" field.

4.7.4. Enter next item to search.

- Human Resource Records can be requested by: Human Resources and Directors
- Director Records can be requested by: Directors
- Medical Records can be requested by: Human Resources, Directors and the Lab Nurse

4.8. Record Requests and Charge-Outs

4.8.1. There is a 30-day charge-out period for all records. The person who has charged out a record is responsible for that record until it is returned to The Records Department.

4.8.2. Records can be requested by stopping by, email, or calling the Records Department. A request for records must include the Lab ID, ascension number, or record title including the year at a minimum. It is preferable to also include the client name and mail date.

4.8.3. Records that are stored off-site can be retrieved and delivered on Tuesday and Thursday. In an emergency, records can be retrieved from off-site storage within one hour at a cost of $40.00.

4.9. Vital Records

4.9.1. SQL Server Backup Policy

4.9.1.1. The primary tool used for the backup of the SQL, Office and Target DB Servers will be Backup Exec for Windows NT (including the Agent for Microsoft SQL Server for NT). This will allow a singular backup format and strategy for the server's operating system, data files and databases.

4.9.1.2. The backups are scheduled as follows:

Scheduled Job	Job Type	Frequency	Retention Period	Storage Location
Monthly	Full (including OS, data, and databases) – SQL and Office servers only	First Monday of Month 1:00 AM	1 Year	On-site Fire Safe
Weekly	Full (including OS, data, and databases) – SQL and Office servers only	Every Monday 1:00 AM	4 Weeks	On-site Fire Safe
Weekly	Full (including OS, data, and databases) – Target DB servers only	Every Monday 1:00 AM	Indefinitely	On-site Fire Safe

Daily	Differential (OS and data) Full (including OS, data and databases) – Target DB only Full (including OS, data, and databases) – Target DB servers only	Tuesday-Friday 1:00 AM	1 Week	On-site Fire Safe

4.9.1.3. The backups should be configured as follows:

4.9.1.4. SQL, Office Servers and Target DB

4.9.1.5. Server Backup Media Set Should have Overwrite Period = 7 day, Append Period = Infinite.

4.9.1.6. Job Properties

4.9.1.6.1. Append to media; overwrite if no appendable media is available.

4.9.1.6.2. Device – Quantum 0

4.9.1.6.3. Media Set – Server Backups

4.9.1.6.4. Write Checksums to Media (True)

4.9.1.6.5. Compression – Hardware (if available, otherwise software

4.9.1.6.6. SQL – DATABASE – Backup Entire Database

4.9.1.7. Backup Method for Files

4.9.1.7.1. Full Backup – NORMAL – Back Up Files – Reset Archive Bit

4.9.1.7.2. Differential Backups – DIFFERENTIAL – Change Files

4.9.1.8. Backup Selections

4.9.1.8.1. Include all data directories

4.9.1.8.2. SQL Server Databases include only non-QuantIMS objects of DATAMIRROR, include all other databases except PUBS and NORTHWIND.

4.9.1.9. The monthly backup tape consists of the first Monday of the month of the weekly backup tapes. There is no additional monthly backup performed.

4.9.1.10. All backups should be monitored and verified by the LAN Administrator. Execution of the backups and tape handling is also the lab's LAN administrator's responsibility.

4.9.1.11. Once the tapes are brought to records, they are logged in on SQL Backup Tapes Logsheet and then returned to be recycled.

4.9.2. Blueprints

4.9.2.1. The building blueprints have been duplicated on aperture cards, which are stored off-site, in a vault at Cintas Records Management. A copy is kept on-site in a fireproof cabinet.

4.10. Record Retention

4.10.1. Records are retained for a total of five years unless a client specifies other retention requirements. Drinking water analyses are kept ten years, and NELAP requires five years

4.10.2. In the event that the laboratory transfers ownership or goes out of business, the records will be maintained until the scheduled retention period has been met. Clients with contractual agreements for the return of their records will be contacted. Those records will be returned at the client's expense. Proof of ownership and responsibility will be required before the records may be released.

4.10.3. This procedure ensures compliance with the STL Record Retention Policy, P-L-001.

4.10.3.1. Special Record Retention Requirements

Program	Retention Requirement
Ohio – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement and others as negotiated.
Ohio Voluntary Action Program	10 years

4.10.4. At the end of the specified retention period, records are destroyed per criteria in SOP NC-QA-0013, Inventory/Warehouse Control.

5. DEFINITIONS

5.1. Refer to the glossary in the STL North Canton Laboratory Quality Manual (LQM), latest version.

5.2. Chromatogram(s) refers to raw data generated from laboratory organic analyses.

5.3. Field - one part of record that contains specific information. (Example: the box number would be one field)

5.4. Record - consists of a number of information fields. (Example: the box number, location and department would make up a record)

5.5. Batch - one or more boxes entered for a single department.

5.6. Accession number - six digit bar code number used to identify a single box.

5.7. File boxes - the standard box used is 1.2 cubic feet in size and weighs approximately 35 - 40 pounds full.

5.8. Vital records - records that are essential to protecting the assets of the company and its ability to continue operations are considered vital.

5.9. References

5.9.1. S-Q-001, Official Document Control and Archive, latest version

5.9.2. P-L-001, Record Retention, latest version

5.9.3. NC-QA-0013, Inventory/Warehouse Control, latest version

6. APPENDICES

6.1. Example Records Transfer Form

6.2. Department Codes for Report Tracker

Appendix 1: Example Records Transfer Form

RECORD TRANSFER FORM

Directions: 1) Complete one form per box and detail contents as much as practical. 2) Write record title on side of the box with a handle and year of content. 3) Send Record Transfer Form approved and signed by Department Record Liaison to records. 5) After receiving barcode label attached under the box handle. 6) The location, which is to be written on the right side of box, (same side with handle) and circled. 7) Place Record Transfer Form in box. 8) Place box in centralized record pick-up area.

NAME: **DATE: (RELEASED BY)**

Record Series Number: ***0069 – QUAL ASSUR*** **Department Code:** ***00QA – Quality Assurance***

Record Title:

(No more than 50 Characters) (Example: 2000 Sample Removal Requests, January 2000 Batch QC, etc.)

Date of Records: **Retention Period:** **Destruction Date:**

(One year per box ONLY) (Refer to Retention Policy, LEG-004) (Year Only)

Matrix: Paper*X* **Film/Fiche** **Diskette** **Mag Tape** **CD** **Other**
Record Liaison:

Accession Number: **Box Location:**

Records Use Only:

Batch#: _____ **AS400 entry:** _____ **Report Tracker entry:** _____

Contents: (Be very specific. List when applicable: client code, lot number, name of client, batch numbers, instrument number, month date range, etc.)

Appendix 2: Department Codes for Report Tracker

Code	Department	Code	Department
00HR	Human Resources	0MSS	GC/MS Semivolatiles
00IS	Information Services/Technology	0MSV	GC/MS Volatiles
00PM	Project Management	0WET	Wet Chemistry
00QA	Quality Assurance	MAIN	Maintenance
0ADM	Administration	OPER	Operations
0EHS	Environmental Health & Safety	PURC	Purchasing
0EXT	Organic Extractions	RMGT	Records Management
0FAS	Field Analytical Services	SALE	Sales
0GCS	GC Semivolatiles	SAMC	Sample Control
0GCV	GC Volatiles	SHIP	Shipping
0LAW	Legal	TRAI	Training
0MET	Metals		

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SOP No. NC-QA-0018, Rev. 8
Effective Date: 02/15/07
Cover Page

Title: Statistical Evaluation of Data and Development of Control Charts

[Method: None]

Approvals (Signature/Date):

Dorothy J. Leeson 12/13/07
Quality Assurance Manager Date

[signature] 12/14/07
Laboratory Director Date

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Technical Director Date

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STL STANDARD OPERATING PROCEDURE

TITLE: STATISTICAL EVALUATION OF DATA AND DEVELOPMENT OF CONTROL CHARTS

(SUPERSEDES: REVISION 7, DATED 12/08/04)

Approved by: Dorothy J. Leeson — Quality Assurance Manager — 3/20/07 Date

Approved by: [signature] — Laboratory Director — 3/14/07 Date

Approved by: Mark Bruce — Technical Director — 3/13/07 Date

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1. PURPOSE

1.1. The purpose of this SOP is to describe the requirements for: (1) statistically establishing QC acceptance criteria and (2) long-term trend analysis of QC data using control charts at the STL-North Canton Laboratory.

1.2. The control chart is an effective tool for long-term trending because it records in real time the accuracy (bias) and precision of the appropriate parts of the measurement process. The control chart provides the means to demonstrate statistical control.

1.3. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory.

2. RESPONSIBILITIES

2.1. Analyst

2.1.1. All QC data is entered into the Laboratory Information Management System (LIMS) for statistical evaluation for the generation of control charts. (Data entry may be automated or may be completed from the report generation software as part of the report or review group activities.)

2.1.2. Monitor method performance using established limits and identify any out-of-control situation. Respond to out-of-control conditions. QC Data results are considered out of control when recoveries exceed established control limits.

2.2. Group Leader/Supervisor

2.2.1. Respond to out-of-control conditions.

2.3. QA Department Staff

2.3.1. For analytical methods, coordinate updating of control limits. During this process, review control charts to detect any trends in routine analytical procedures.

2.3.2. Archive control charts and statistically derived QC acceptance data.

2.3.3. Publish statistically derived QC acceptance criteria.

2.3.4. Provide guidance in the development of control charts and in the application of QC samples and acceptance data.

2.3.5. With the Laboratory Director, ensure that Operations staff conforms to the requirements provided in this SOP.

2.4. Project Manager

2.4.1. Incorporate updated limits into project-specific QAPPs.

2.4.2. Submit project-specific control charts to the requesting client.

3. SAFETY

3.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

3.2. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in the laboratory area, appropriate personal protective equipment and precautions must be utilized.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor and the EH&S Coordinator.

4. PROCEDURE

4.1. Empirical Establishment of QC Acceptance Limits

4.1.1. The assessment of QC sample data shall be performed by comparing precision and accuracy results against control limits. As defined in the following subsections, the control limits used for this comparison shall be either in-house (statistically generated using historical data) control limits or published limits from methods, contracts, or project QA plans.

4.1.2. In-house limits for all QC data must be determined and compared to those limits published in the methods for applicable matrices. Method limits will be employed until sufficient QC data are acquired. A minimum of 20 to 30 of the most recent data points should be used to establish in-house limits based on historical performance data for each major method. Periodically, QC data may need to be reviewed and house limits reestablished whenever a <u>significant</u> <u>change</u> in an analytical process occurs.

4.1.3. Control limits shall be generated for each matrix (i.e., aqueous and soil) for preparative methods, using data from at least 20 of the most recent data points. Limits are generated using a six-month timeframe to ensure the minimum number of points are included.

4.1.4. In-house control limits shall be established for the following samples:

4.1.4.1. Laboratory control sample (LCS) spike recoveries for method required analytes list.

4.1.4.2. Matrix Spike and Matrix Spike Duplicate (MS/MSD) spike recoveries for method required analytes list.

4.1.4.3. Surrogate spike recoveries in LCSs for organic analyses only.

4.1.5. Control limits shall be established for all methods unless specified in project plans such as the Louisville Chemistry Guideline (LCG) or the DoD QSM.

4.1.6. The calculations used to generate the control limits for accuracy (%R) are described in the following subsections.

4.1.6.1. The %R is defined as the observed concentration in LCS divided by the theoretical concentration of the spike or LCS, times 100:

$$\%R = \frac{Found}{True} \times 100$$

4.1.6.2. The mean percent recovery and standard deviation is calculated using the following formulas:

$$\overline{\%R} = \frac{\sum_{i=1}^{n} \%R_i}{n}$$

$$S = \sqrt{\frac{\sum_{i=1}^{n} \left(\%R_i - \overline{\%R}\right)^2}{n-1}}$$

where:

%R	=	the mean percent recovery
$\%R_i$	=	the percent recovery of an LCS
n	=	the number of data points
S	=	the standard deviation of the data set of percent recoveries

4.1.6.3. The warning (95% or 2-sigma) and control limits (99% or 3-sigma) are then calculated from the following equations:

$$\text{Upper Control Limit} = \overline{\%R} + 3\,s$$

$$\text{Lower Control Limit} = \overline{\%R} - 3\,s$$

$$\text{Upper Warning Limit} = \overline{\%R} + 2\,s$$

$$\text{Lower Warning Limit} = \overline{\%R} - 2\,s$$

where:

$\overline{\%R}$	=	the mean percent recovery
S	=	the standard deviation of the data set

4.1.7. Control limits will be recalculated after excluding the following points from the calculations:

4.1.7.1. Samples with values outside control limits due to assignable cause.

4.1.7.2. True outliers as defined in the Grubbs test.

4.1.8. The LIMS is equipped to perform a Grubbs outlier test used to generate the control charts.

4.2. Control Chart Generation – QA Access

4.2.1. A control chart (X chart) is generated using the LIMS or a commercially available software to monitor accuracy and precision by plotting the LCS %R data in a graphical format as follows:

4.2.1.1. The average of the %R determinations for the original data set is established as the midpoint on the Y axis of the graph.

4.2.1.2. The upper and lower warning and control limits are plotted as solid horizontal lines across the graph at their respective points on the Y axis.

4.2.1.3. The calculated %R of each spiked sample is plotted chronologically on the graph to determine whether the recovery is within the warning and control limits of the control chart.

4.2.2. Control charts can be generated from any networked system (see network administrator) as follows:

4.2.2.1. From the Desktop, TraQAr double click the "Control limit " icon button.

4.2.2.2. Select "QA Access"

4.2.2.3. Select "location" (North Canton), "QC Type" (LCS/DCS, MS/MSD, LCS/DCS Surrogates, MS/MSD Surrogates, All Surrogates), "Start and End Date" (default is six months), and then spike list number.

4.2.2.4. Select 'QuantIMS Spike List"

4.2.2.5. Select "QC Program"

4.2.2.6. Select "Collect Data". This screen will run and then the Control Limits Review screen comes up.

4.2.3. Control Limits Review screen

4.2.3.1. A "Grubbs test complete" sign comes up, select OK

4.2.3.2. A minimum of 20 points are required. If points are below 20, select close form and increase the start and end date to no more than one year.

4.2.3.3. If there are at least 20 points, select "Control Limits Report". Right click on report and select print.

4.2.3.4. Select "Control Charts Report". Right click on report and select print.

4.2.3.5. This procedure demonstrates both "before" and "after" data sets used for control charts generation.

4.2.4. The following information must be present on the control charts or in an associated table:

4.2.4.1. Parameter, Analytical Method and preparation procedure

4.2.4.2. LCS Batch ID allowing cross-reference to LIMS containing all analytical information.

4.2.4.3. Matrix

4.2.4.4. Number of points used

4.2.4.5. Mean

4.2.4.6. Standard Deviation

4.2.4.7. Percent recoveries

4.2.4.8. Upper and Lower warning and Control limits

4.2.4.9. Chart generation date.

4.2.4.10. An example of the report is provided in **Appendix A.**

4.3. Evaluation of Control Charts

4.3.1. Criteria for an Out-of-Control Conditions

4.3.1.1. The causes for a shift or a trend in control charts could result from many reasons, including, but not limited to:
(1) incorrect preparation of a standard or a reagent,
(2) sample contamination,
(3) improper storage or preservation,
(4) incorrect instrument calibration,
(5) poor analytical technique, and
(6) deviation from the analytical method.

A measurement process for a particular analyte will be considered out-of-statistical-control when one of the following conditions occur:

4.3.1.1.1. A single point outside 3-sigma control limits

4.3.1.1.2. A series of eight consecutive points on the same side of the central line.

4.3.1.1.3. A series of three consecutive points between the warning limits and control limits.

4.3.1.1.4. A series of six consecutively increasing or decreasing points on the same side of the centerline.

4.3.1.1.5. A cyclic pattern of control values.

4.3.1.1.6. These conditions may indicate that the measurement system is out of statistical control. When this situation occurs, the data must be evaluated thoroughly to identify the most appropriate corrective action to be implemented. The problem and its solution may be documented through a Nonconformance Memo as appropriate. Exceeding warning limits will only require a close observation of the measurement system. In reviewing control charts, any significant changes in key analysts, instrumentation, standard reference materials, or processes must be kept in mind to explain potential out-of-control situations. After thorough evaluation of the data and documentation of corrective actions taken, the QA Department must determine if the defensiveness of analytical results generated during the out of control situation has been jeopardized. If it is determined that data defensiveness has been compromised, the client will be notified of the out of control situation.

4.4. On-Line Control Chart Generation

4.4.1. Laboratory group leaders or designees have access to the TraQar On-Line Control Chart program. This feature is used to view results in real time to determine trends or corrective actions.

4.4.2. From the Desktop, TraQAr double click the "Control limit " icon button.

4.4.3. Select "On-line Control Charts."

4.4.4. Select the QC Type and Group from the drop down menus.

4.4.5. The start and end date are automatically set for 30 days. The date can be changed as needed.

4.4.6. Select the appropriate Method from the list.

4.4.7. Select the appropriate QC code. In most cases, this will be 01. Then choose the appropriate spike list.

4.4.8. Press the Collect Data button.

4.4.9. A screen with Chart Options will appear. The default choices are All Analytes, All Instruments, and All Matrices. These choices may be modified as needed.

4.4.10. Select OK. Control charts will appear on the screen for each analyte and matrix. The report can be printed or viewed on line.

4.4.11. Refer to Section 4.3 for Control Chart Evaluation.

4.5. Setting Control Limits

4.5.1. The working control limits to be used by the laboratory are based on evaluation of the calculated laboratory statistical performance and available interlaboratory limits provided in the reference methods. Note that some SW-846 methods only supply single-operator or single-laboratory method performance data, which may not be appropriate.

Accuracy Evaluation:

Lower Limit Evaluation	Upper Limit Evaluation	Accuracy Decision
Laboratory-generated Lower Limit > Guidance Limit	Laboratory-generated Upper Limit > Guidance Limit	Use laboratory-gen. Lower Limit & Guidance Upper Limit
Laboratory-generated Lower Limit > Guidance Limit	Laboratory-generated Upper Limit < Guidance Limit	Use laboratory-gen. Lower Limit & laboratory-generated Upper Limit
Laboratory-generated Lower Limit < Guidance Limit	Laboratory-generated Upper Limit > Guidance limit	Use guidance Lower Limit & guidance Upper Limit
Laboratory-generated Lower Limit < Guidance Limit	Laboratory-generated Upper Limit < Guidance limit	Use guidance Lower Limit & laboratory-generated Upper Limit

Precision Evaluation:

Range Evaluation	Precision Decision
Laboratory-generated precision value > Guidance precision	Use guidance precision
Laboratory-generated precision < Guidance precision	Use laboratory-generated precision

Notes: If the decision is to use guidance limits from the method, the laboratory should investigate procedural improvements leading to better performance.

The following outlines other criteria:

Min/Max	Limit	Percentage	Inorganics	Organics
Minimum	LCL	10%	√	√
Maximum	LCL	90%	√	√
Minimum	UCL	110%	√	√
Maximum	UCL	199%	√	√
Minimum	RPD	20%	√	
Maximum	RPD	99%	√	√
Minimum	RPD	30%		√

5. DEFINITIONS

5.1. Control Chart - A graphical QC tool to monitor method performance over time and to establish acceptance limits.

5.2. Relative Percent Difference (RPD) - A measure of intra-lab precision based on a duplicate sample analyses.

5.3. Grubbs Test – Extension of sample sizes and percentage points for significant tests of outlying observations - a statistical outlier test.

5.4. Percent Recovery (%R) or Recovery - A measure of the accuracy (bias) of the measurement process based on a comparison of a measured value for a fortified (spiked) QC sample against the known spiked values.

5.5. Precision - A measure of mutual agreement (or variability) among individual measurements of the same property, usually under prescribed similar conditions.

5.6. Accuracy - The degree of agreement of a measurement (or an average of measurements of the same thing) with an accepted reference or true value. Accuracy is the measure of bias inherent in the system.

5.7. Bias - A systematic (consistent) error in test results. The difference between the population mean and the true or reference value, or as estimated from sample statistics; the difference between the sample average and the reference value.

5.8. X-chart – A control chart that plots a single measurement of a property (e.g., percent recovery) of quality control samples over time. The chart consists of a single line that is the mean of the statistic, warning limits at ± two standard deviations, and control limits at ± 3 sigma.

5.9. Assignable cause – A known reason for an outlying result (e.g., no spike added).

5.10. Duplicate – A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

5.11. Laboratory Control Sample (LCS)

5.11.1. Organics - A LCS is a volume of deionized laboratory water (for water samples) or a suitable solid material (e.g., clean sand) (for soil/sediment samples) which is spiked with compounds of interest and subjected to the entire analytical procedure in order to estimate the accuracy of the method via percent spike recovery.

5.11.2. Inorganics - A well characterized liquid or solid sample which is prepared, digested or extracted along with each analytical batch of samples.

6. REFERENCES

6.1. STL Quality Management Plan (QMP), current revision.

6.2. STL Laboratory Quality Manual (LQM), current revision.

6.3. Test Methods for Evaluating Solid Waste, Third Edition, SW-846, US EPA, Final Update III, December 1996.

6.4. QA Policy, QA-003, current version.

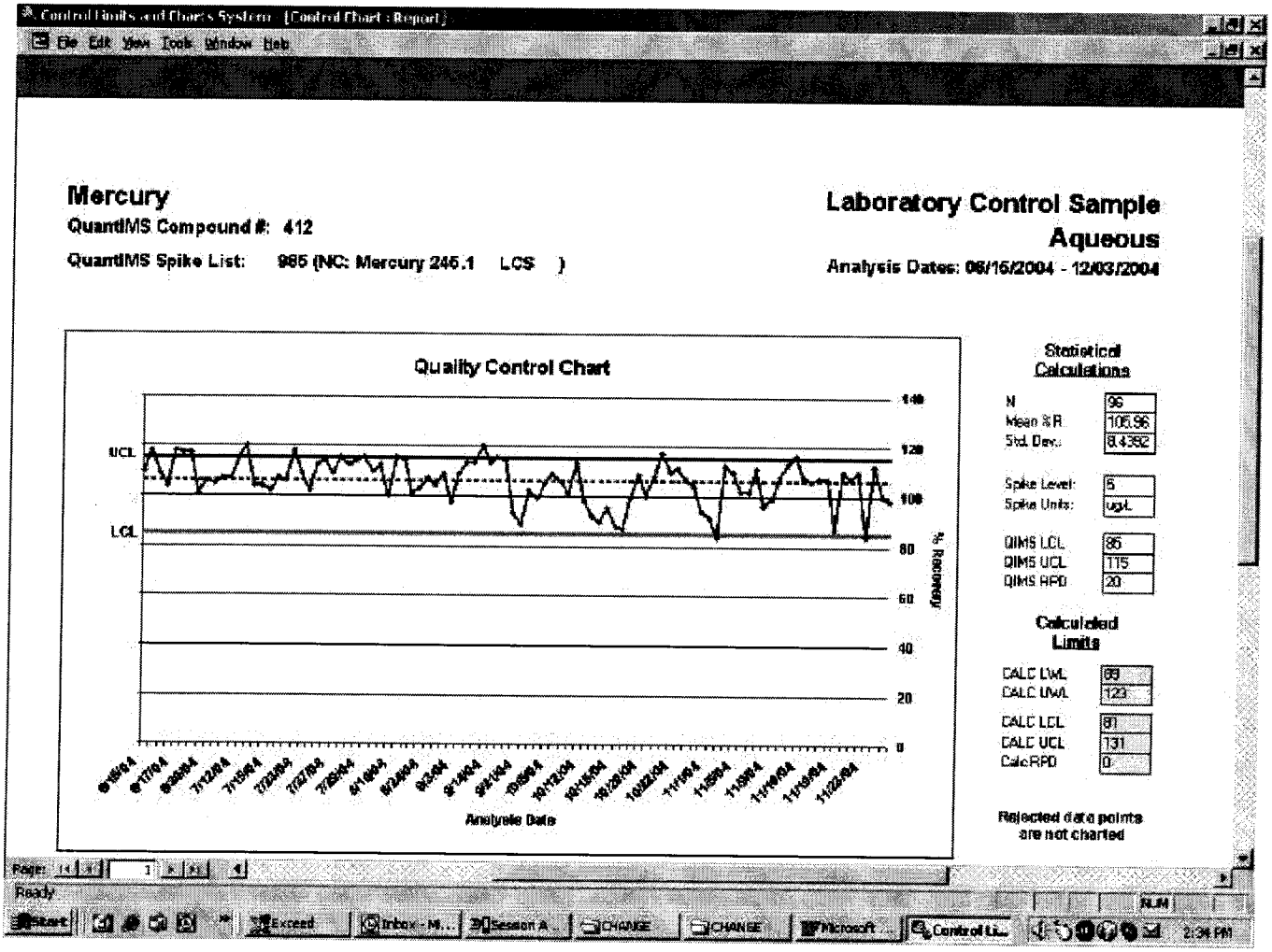
6.5. Supplemental Practices for DoD Project Work, NC-QA-0016, current version.

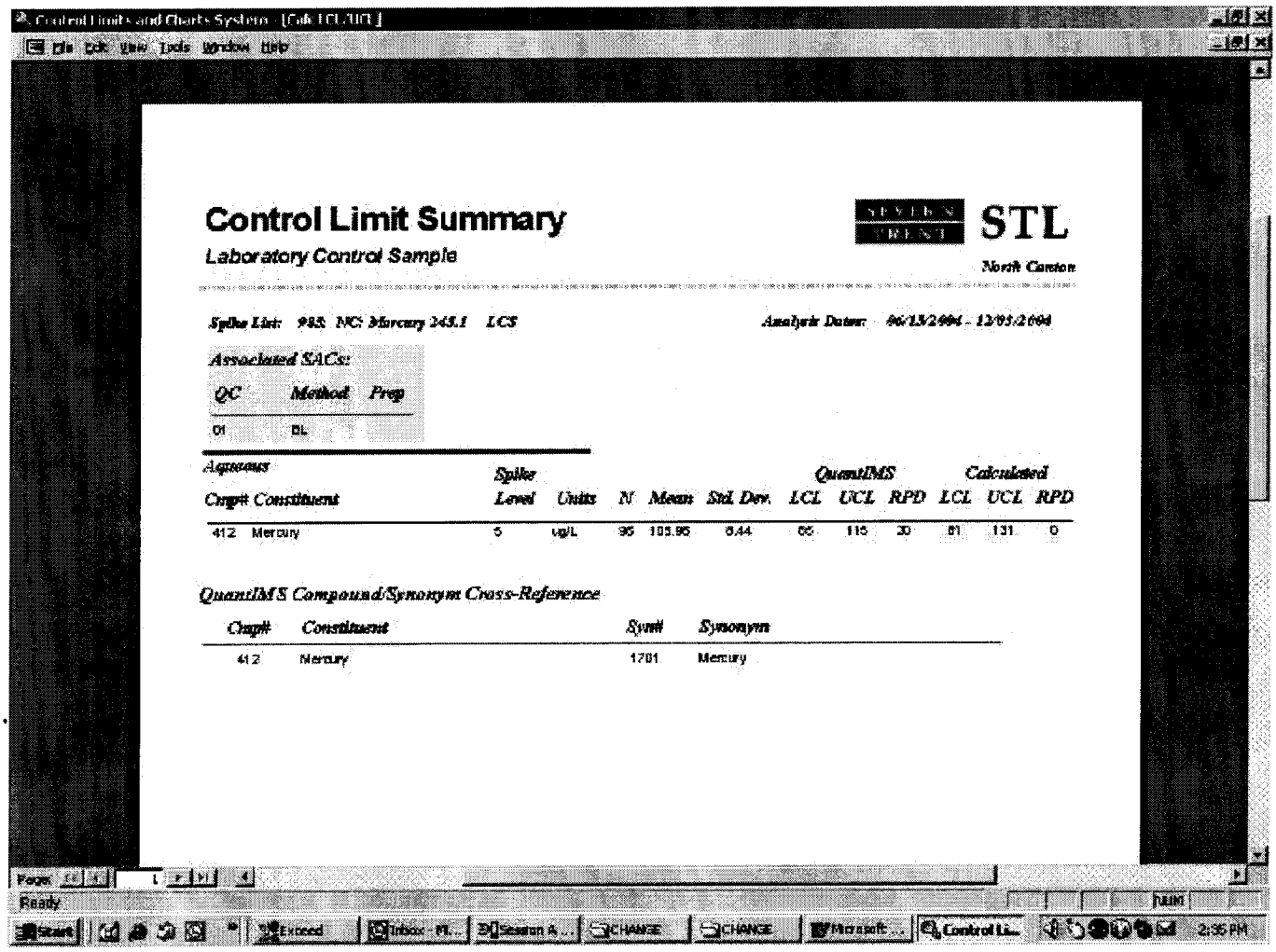
6.6. Dept. of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006 and future revisions.

6.7. Louisville Chemistry guideline (LCG), Version 5, June 2002.

APPENDIX A

Examples of Control Charts





Control Limit Summary

Laboratory Control Sample

STL
North Canton

Spike List: 985 NC Mercury 245.1 LCS

Analysis Dates: 06/15/2004 - 12/05/2004

Associated SACs:

QC	Method	Prep
01	BL	

Aqueous

Cmp#	Constituent	Spike Level	Units	N	Mean	Std. Dev.	QuantIMS LCL	QuantIMS UCL	QuantIMS RPD	Calculated LCL	Calculated UCL	Calculated RPD
412	Mercury	5	ug/L	95	105.95	8.44	85	115	30	81	131	0

QuantIMS Compound/Synonym Cross-Reference

Cmp#	Constituent	Syn#	Synonym
412	Mercury	1701	Mercury

Revised by CF1, 10/16/06
Reviewed 12/11/06

Revised by CF2, 12/26/06
Revised by CF3, 3/12/07

Title: Sample Receipt and Procedures

Approvals (Signature/Date):

1/17/2008 Mike Flournoy Date Technical Manager	1/17/08 Joe Schairer Date Health & Safety Manager / Coordinator
1/17/08 Pamela Schemmer Date Quality Assurance Manager	1/17/08 Karla Buechler Date Laboratory Director

This SOP was previously identified as SAC-QA-0003.

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1. PURPOSE

1.1. This SOP describes the procedures for laboratory chain-of-custody, including receipt and acceptance of sample shipments, storage requirements, generation of computer records, and corrective actions for sample receipt anomalies.

2. RESPONSIBILITIES

2.1. It is the responsibility of the employee to perform the procedure described here in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and Departmental Supervisors of this facility to ensure that the analysis is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

3. DEFINITIONS

3.1. Refer to Glossary in the STL Sacramento LQM.

3.2. Legal chain of custody: Based on client request, legal chain of custody may be generated to support litigation. Legal chain of custody are generated per the Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth edition, January 2005, Appendix A.

4. INTERFERENCES

4.1. Any checks on samples, or storage of samples, should be done to eliminate any cross contamination.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported immediately to a supervisor, the EH&S Staff, or a senior manager.

5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide sufficient protection when handling closed sample containers.

5.2.2. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred, sub-sampled and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.2.3. Laboratory procedures such as repetitive use of pipettes, repetitive subsampling, moving heavy shipping containers, unloading shipping containers, and manipulation of glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2.4. Safety policies apply to ALL sample administration visitors, including auditors, employees, couriers or clients who deliver samples.

5.2.5. Some types of biological samples may present special hazards. Refer to Appendix 11 of this document for more information.

5.2.6. Samples containing or potentially containing chemical warfare agents or degredates present a special hazard. Review Appendix 10 of this document before opening any coolers containing these types of samples.

5.3. PRIMARY MATERIALS USED

5.3.1. The following is a list of the materials used to preserve samples that are received in sample administration, which have a serious or significant hazard rating. Samples arrive at sample administration preserved in the field; these materials are not found or used in the sample administration area outside of samples. NOTE: This list does not include all hazards that may be present in samples. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. 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
Sulfuric Acid (1)	Corrosive Oxidizer Dehydra-dator	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.
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.
Hydrochloric Acid (1)	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.
Isopropyl Alcohol	Flammable	400 ppm - TWA	Flammable liquid and vapor. Inhalation of vapors or ingestion can have narcotic effect, with dizziness, drowsiness, and headache. Exposing eyes to either vapors or splashed liquid can result in severe irritation, corneal burns and eye damage.
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous. 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.
Sodium Bisulfate	Corrosive	None listed	Contact may cause skin/eye burns. Inhalation can cause irritation of the respiratory tract with burning pain in the nose and throat, coughing, wheezing and shortness of breath. Causes chemical burns to the respiratory tract. May cause fatal spasms, inflammation or pulmonary/respiratory edema.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. IR thermometer calibrated at a minimum of once per quarter against an NIST reference.

6.2. Filament thermometer calibrated at a minimum of once annually against an NIST reference.

6.3. Probe thermometer capable of reading to 0.1°C calibrated at a minimum of once annually against an NIST reference.

6.4. pH paper (Range pH 2 to pH 12 or equivalent)

6.5. Pipette

6.6. Calibrated balance capable of reading 0.1g

6.7. Packing material including wet ice

7. STANDARDS AND REAGENTS

7.1. Not applicable. Use of preserves or other reagents is performed in laboratory areas.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. This SOP does not address sample collection.

8.2. Preservation and storage of samples is determined by each method. See method SOPs, and section

9. QUALITY CONTROL

9.1. Not applicable.

10. CALIBRATION

10.1. Thermometers are calibrated according to SAC-QA-0016.

10.2. All electronically operated thermometers (including IR thermometers) must have their calibration verified each day of use (see SAC-QA-0016, Thermometer Calibration for further details). IR thermometers are to be calibrated quarterly against a "NIST" reference.

11. PROCEDURE

11.1. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described (see SAC-QA-0023, Nonconformance and Corrective Action Systems for further details).

11.2. Before samples are received, the laboratory should provide the client's sample collection personnel the STL Sacramento Sample Receiving Acceptance Policy (see appendix 1). It is sent along with the bottle order.

11.3. Receiving shipment

11.3.1. Verify accuracy of each shipping container's delivery address. Note if the custody seal number is present and the seal's condition.

11.3.2. Record each delivery received in the computer entry SRL (Sample Receipt Logbook), see appendix 2 for screen-print. A single shipping container may be listed as one or several lots. Record the client and/or shipper name(s), receipt date and time. If the lot number is generated for the shipment at this time, record the number in the far-left column. If a single shipment is subdivided into multiple lots at a later time, all lot numbers are listed on the original logbook entry. In the event of multiple package deliveries, the receipt time is the carrier's time of arrival, not the time when each package is handled. This document is required for legal chain of custody.

11.3.3. Initiate a LRC (Lot Receipt Checklist) for each sample shipment (see appendix 3). Each person who completes an entry must also enter initials and dates in the corresponding columns. For non-applicable items, enter "N/A", with initials and dates. The initiator must complete entries A, B, and F-K, unless responsibility for the shipment is immediately passed to another technician.

11.3.3.1. Item A: Enter client and assigned PM.

11.3.3.2. Item B: Enter SRL number as generated by the SRL program.

11.3.3.3. Item F: Enter date shipment is received and initial.

11.3.3.4. Item G: Enter time shipment is received.

11.3.3.5. Item H: Enter carrier (method of delivery). "Over the counter" indicates a delivery made directly to the laboratory

by a client.

11.3.3.6. Item I: Enter condition of custody seal (if present). Enter pre-printed custody seal number or "N/A". Custody seals are defined by the presence of the shipper's signature accompanied with date/time. Note the presence of custody tape (which will have no space for entries and crumbles when removed). This is also required for legal chain of custody.

11.3.3.7. Item J: Record ownership/disposition of shipping container. Non-company containers must be returned to client if possible.

11.4. Opening shipment

11.4.1. Following safety policies, open the shipping container and remove paper work. Bechtel, Navy Clean or Clean 3 projects must all be opened in the hood. In addition, shipments accompanied by MSDS documentation, shipments of concentrated product, or exhibiting strong, noxious odors must be opened in the hood. If a shipment contains broken or leaking samples, place the container in fume hood and notify the project manager via e-mail. Digital pictures may be taken of the broken samples and the files attached to the project manager's email. Dispose of broken samples according to the facility hazardous waste procedures. The Hazardous Waste Specialist or Environmental Health and Safety Coordinator should be contacted if additional information is required.

11.4.2. Assume custody of the samples by signing and dating the COC in the section marked "Received for Lab By" or "Received By". This is also required for legal chain of custody. Immediately after opening, ascertain if ice or artificial coolants are present. Measure temperature and record results under item K of the LRC.

11.4.2.1. Use the infrared thermometer by directing the thermometer at sample containers making sure no labels or packaging materials are interfering with the direct contact of the infrared beam and the sample container. If the COC lists a temperature blank, or one is present in the shipping container, locate the blank and measure the temperature. Take temperatures of random samples in the cooler to determine the average temperature. Measure temperature and record results under item K of the LRC.

11.4.2.2. A non-conformance memo must be entered if the temperature reading is below 2°C or above 6°C. This notifies the project manager via email and provides a hard copy to include with the lot folder. In the non-conformance memo software, be sure to include the client ID for all samples that are associated with a temperature exceedance.

Note: Wisconsin compliant samples must be maintained at temperatures ≤4°C.

11.4.2.3. Some sample matrices do not require cooling during transit. Situations where acceptable temperature range exceedance may be expected include, but are not limited to:

- Samples delivered within six hours of close of daily sampling event; a non-conformance memo must be filed in this case, indicating sampling and receipt time.
- Paperboard and dry pulp samples; non-conformance memo not necessary.
- Dry incinerator ashes; non-conformance memo not necessary.
- Samples for metals analysis only; non-conformance memo not necessary. Note: This does not apply to soil samples for mercury analysis. Such samples must be shipped on ice at 4 °C, and narrated with a non-conformance memo if not on ice.
- Dry product samples; non-conformance memo not necessary.

11.4.3. Verify that the sample collector's name appears on the COC. Record the result under item K. Record the pre-printed COC number (if a COC is present) and the sequential document number under item K of the LRC. Examine accompanying documentation. If documentation is absent and the shipment arrival was unexpected, contact the lead project manager for assistance.

11.5. Prioritizing workload

11.5.1. Examine the analyses requested and the sampling dates/times. Be aware of any notation for expected Turn-Around-Time (TAT), or a due date that is less than 14 days, or hold times that will expire within 72 hours. Most aqueous organic analyses require extraction within seven days of sampling. If three days or less are remaining, consider the lot to be a priority and file in a manila folder (Methods 8280 and 8290 dioxin/furan

analyses are an exception, requiring extraction within thirty days).

11.5.2. Any analysis that has a standard holding time of forty-eight hours or less is considered a "Short-Holding Time" (SHT) analysis. These are reported to the affected department immediately with the Short Holding Time Test notification (see appendix 4). The receiving chemist initials and dates item O of the LRC. Any requested TAT of fourteen days or less is also considered a priority.

11.5.3. Prioritize the shipment according to the TAT, while ensuring extraction holding times have not been exceeded. "RUSH" projects are placed in a red folder, SHT analyses are processed immediately, projects requiring shipment of subcontracted out samples are placed in purple folders, projects requiring project manager's immediate attention are placed in blue folders while all other projects are placed in manila folders. If a project with a short TAT also includes SHT analyses, file in a red folder.

11.6. Sample processing

11.6.1. Select the project with the highest priority:

1. Short holds are processed immediately.
2. Blue folder; (requires project manager's immediate attention)
3. Red folder; (rush)
4. Purple folders; (to be shipped out)
5. Manila folder; (normal TAT)

11.6.2. Read shipment documentation. Ideally, samples are to have possession documented on a COC form. The COC will identify samples individually by alphanumeric designators, list sampling dates/times for each sample, requested analyses and document possession. Signatures of possession qualify samples as having been "received under Chain-of-Custody". A Letter-of-Transmittal is also accepted as definitive documentation. Other forms of documentation include Request for Analysis, Shipping Order, Purchase Order, and various computer listings of sample information. If no documentation that lists sample identifications exists, complete a COC when accepting the samples.

11.6.3. Check the samples against the COC for accuracy (e.g., sample ID, collection date/time, etc.). If the samples have not been received in good

condition, it must be noted on the LRC and an NCM must be filed. All discrepancies must be noted as well, including the lack of a relinquishing signature from the shipper.

NOTE: Good condition is loosely defined as all containers intact with no obvious discrepancies present. The shipment is estimated at this time to be viable; what is being requested coincides with what has been received. Temperature exceedances and minor discrepancies become issues when so specified by contract or client instruction. The presence of bubbles in volatile containers is documented. All such observations must be documented on custody chains, the LRC (item Q), and by using the non-conformance memo software.

11.6.4. Complete the bottle inventory on the project receipt checklist (see appendix 5). Compare the containers and their state of preservation with the list of analyses requested. Although some exceptions are permitted regarding particular clients, container types must compare to those listed in the STL Sacramento Laboratory Quality Manual tables 8.5-1, 8.5-2 and 8.5-3. Discrepancies of this nature must also be annotated on the COC and a non-conformance memo filed. Complete item P and Q.

11.6.5. For all work sampled at or concerning government property, any federal projects, or as specified in client QAPjPs, the pH of the preserved aqueous samples must be checked and recorded, except for the VOA vials. The pH is noted as <2 or >12 with the preservative type following (example: pH<2 H_2SO_4). Complete item L on LRC.

11.6.5.1. After placing the samples in the hood, invert the sample container three times, remove the cap, insert a clean pipette into the sample container and remove a small amount. Release a droplet of the aliquot on a fresh piece of pH indicator paper. Compare against the pH color grid table located on the pH strip container. Do not reuse the pipette. Use a clean pipette for each container. Recheck readings that indicate samples were unpreserved when they should have been. Note discrepancies on the COC. Notify the project manager and file a non-conformance memo for the concerned sample. Transport samples to the general chemistry or metals departments when sample preservation is necessary as a corrective action. Complete item L and O on LRC.

11.6.6. Obtain a lot number and quote number. Complete items C and D of LRC.

11.6.7. Note storage location in item E of LRC. Designate a storage location using the following guide:

11.6.7.1. V - volatile containers awaiting transfer to the VOA group refrigerators.

11.6.7.2. F2 - any samples requiring freezing, including plant/animal tissues;

11.6.7.3. R2 - any samples where secondary containment in Ziploc bags is deemed necessary due to smells, spillage, degradation of container exterior or suspected high concentration of analytes.

11.6.7.4. C1 - dry pulp or paper samples;

11.6.7.5. EPA1 - soil samples received from the Environmental Protection Agency, solvents, air toxics projects for method 29 or clients requiring dioxin work under SOW DFLM01.1.

11.6.7.6. WR1/WF1 - refrigerator/freezer storage dedicated to explosives & specialty chemicals projects.

NOTE: Location selections (specify shelf by letter, Example: W4A).

NOTE: Dioxin soil samples received in clear glass jars must be stored in boxes to protect from light.

11.6.8. Label sample containers. Be certain the labels adhere to containers. Attempt to leave all client label information exposed. However, when impossible to do so, affix label so that at least the client ID, sampling date/time, and preservative are showing. Preprinted laboratory names may be covered with no consequence. If label adhesive is insufficient, use cellophane tape to secure label. Place sample in Ziploc bag when storage location is R2. Initial/date the LRC item M. Labeling may be peer reviewed whenever the complexity of the project warrants a second check to ensure accuracy. A peer compares the order of the samples to the documentation and ensures that all containers of a sample have the same sample number and are sequential. The reviewer then initials/dates the LRC (item N). If no review is performed, the labeler completes item N.

11.6.9. Circumstances where a review is warranted include:

11.6.9.1. A large number of samples are present;

11.6.9.2. Many containers per sample are present;

11.6.9.3. VOA containers are present;

11.6.9.4. Client identifications are illegible or confusing;

11.6.9.5. A review is requested by a STL Sacramento employee;

11.7. Shipping container return

11.7.1. Broken samples and packing material contaminated with spilled samples MUST be disposed of as hazardous lab trash. Shipping containers must be decontaminated before being put back into use. Decontamination procedures will depend on what was spilled. See EH&S staff for specific instructions.

11.7.2. Except when discarded, shipping containers are cleaned in sample administration and either taken to bottle prep or returned to the client. Containers belonging to STL Sacramento are marked with a permanent marker with an easily identified tracking number. If the container belongs to a client, be certain the return address is recorded before stripping the container of used tape and labels. All hazardous materials labeling must **be removed or defaced** in some way. Dry the interior of the container if wet, replace packing and return to bottle prep.

11.7.3. Packing, artificial ice and temperature blanks are returned to the client. Packing which is deemed re-usable may be returned. Packing which resembles trash or is ruined during unpacking is disposed as trash. Drain any water from the container before sealing it closed. Due to partially frozen artificial ice, client containers may not be perfectly dry when returned. Complete a return-mailing label. Secure it with cellophane tape and seal the container.

11.8. Disposal of ice

11.8.1. When ice is present, it is often enclosed in plastic bags. A basin used for dumping ice is located in sample administration. Open the bags and dump ice into the basin. Water should not be left running in the basin as the basin drains into a closed system. When excessive amounts of ice are received, collect the ice in an ice chest and dump it over the storm drains

outside the building. **This is permissible only if the ice and coolers are uncontaminated and the amounts disposed in this way are recorded. Additionally, ice used to ship any soil samples may not be disposed down the storm drain. It may only be disposed of down the basin in sample administration.**

11.9. Lot generation

11.9.1. Lots are created by using quotes that are provided by the project manager. Determine the appropriate quote number by using the "Quote Search" program, via either the client name or project manager. If the quote cannot be determined, send an email to the project manager by using the Review Login Data of the Sample Receipt Logbook program. If the quote information is not available and must be set-up from scratch, determine the name and location of client. This information can be found on the COC, or shipping labels and forward this information to the project manager so they can create a quote.

11.10. Sample login

11.10.1. Refer to the QuantIMS E-Z Login sheet.

11.10.2. Record lot number in SRL on the line corresponding to the shipment's arrival. Complete Lot Review Checklist #1 (see appendix 7). File an HTV, if necessary, by entering "Anomaly" and the lot number at your prompt. Answer the queries, and be certain to mail the HTV to the department supervisor and project manager involved. The HTV is filed by sample administration when a holding time is expired upon receipt. If the holding times have not completely expired for a project, the department handling the analysis in question files the HTV. Place the project manager folder in the project manager's tray and the internal tracking folder in the checkout boxes (both located in sample administration).

11.11. Project manager folder assembly

11.11.1. Place in an appropriately colored folder (see section 5.6.1.):

11.11.1.1. Lot Receipt Checklist #1 and bottle inventory; (yellow page)

11.11.1.2. Lot Review Checklist #1 (green page)

11.11.1.3. Original COC

11.11.1.4. Air bill and secondary documentation

11.11.1.5. Sample confirmation report, (QuantIMS) (white page)

11.11.2. In-house COC folder assembly

11.11.2.1. Align folder (any color) with the tab on right-hand side. On the right-hand side of the folder, staple the in-house COC form (see appendix 6). On the left-hand side, staple a copy of the inventory sheet. If the entire bottle inventory for any sample is not complete on the sample confirmation report, include a copy of the bottle inventory (reverse side of LRC). If an MSDS is provided in shipment, place a copy on the right-hand side of the folder.

11.12. Refrigerator and freezer temperatures

11.12.1. Refrigerator and freezer temperatures will be monitored and recorded once daily Monday through Saturday. Any temperature below 2°C or above 6°C for the refrigerators and above -10°C for the freezer will need corrective action. Refer to SAC-QA-0005, Temperature Monitoring and Corrective Action for Refrigerators and Freezers. Temperatures will be recorded on the appropriate charts. Temperature charts will then be stored in the temperature logbook. All charts for a particular refrigeration unit will be stored together in chronological order.

11.13. Internal sample tracking

11.13.1. Analyst selects sample checkout folder for the lot of interest and ascertains storage location. Analyst removes appropriate containers from storage unit and organizes them by lot and sample number. Analyst completes checkout portion of internal COC. For a legal chain of custody, a sample custodian must verify the record and container labels and initial under the witness field on the internal COC. For non-legal COCs, "NA" may be entered in the witness field.

11.13.2. If a sample is completely used up in the extraction laboratory, then the disposition of the container must be documented. The code DIT (destroyed in testing) may be used. All empty containers not returned to sample administration must be recorded on the Internal COC. If the containers are not to be returned and will enter the waste stream through the extraction lab, the analyst will also enter this information during checkout.

11.13.3. Analyst files the checkout folder before leaving the room. At return, the analyst completes the check-in portion of the Internal COC. Analyst returns samples to their storage location. If subsampling or aliquoting is

performed, whether outside the area or not, containers must be checked out by the analyst. The original containers' whereabouts and those of any other containers generated must be documented. Transfer of volatile containers from VOA to RD and RF do not require verification.

11.14. Sending out subcontracted work

11.14.1. In the event samples require being subcontracted out to another lab, the samples will be processed in the very same manner as all other samples. Samples should be received and processed as described in sections 5.1 to 5.13 of this SOP. The project manager should notify the sample receiving staff as to all samples they require to be shipped out.

11.14.2. Using QuantIMS, generate a new STL Sacramento chain of custody for the subcontracted samples. The destination for the samples should be entered on the COC. Contact the project manager for assistance if any difficulties are encountered.

11.14.3. Log samples to be subcontracted out onto a sample receiving shipping log. In addition, use the in house COC (see appendix 6) to check out the samples from sample control. In the sample check-in section, document that the sample was subcontracted and its' destination.

11.14.4. Samples should be packaged in a manner in accordance with STL Sacramento Receiving Acceptance Policy. Samples are packaged in a plastic bag to insure labels do not get wet. Additional packing may be used to insure samples have adequate protection from breakage. Samples are subsequently packaged in a bag containing copious amounts of wet ice. There should be sufficient ice to maintain the samples within the temperature range of 2°C to 6°C throughout transit. The ice packed samples are stored in the appropriate sized insulated container.

11.14.5. Relinquish custody of the samples by signing the appropriate space on the STL Sacramento COC. A copy should be included with the samples. Insure that it is packed in the shipping container and that it is protected from moisture from ice and or samples (in the event any containers become compromised). The original should be filed in the project manager purple colored folder.

11.14.6. The packed container is then sealed with a generous amount of shipping tape to ensure the contents remain secured. Samples are now ready for shipment. Ensure they are labeled with the appropriate shipping labels and delivered to the courier.

11.15. Client deliveries ("over the counter")

11.15.1. Samples received "over the counter" are samples hand delivered by the client, sampler or courier directly employed by the client's company. The pivotal question in such deliveries is whether the person possessing the shipment is in custodial possession of the samples or merely transporting the shipment. If in custodial possession, the deliverer will need to relinquish custody to you before departing and will probably retain a copy of the documentation. To determine the matter, simply ask the deliverer if they had signed any of the sample documents.

11.15.2. If the client, sampler or courier does not wish to remain while the samples are unpacked and verified, sign the COC in the "Received By" section. Include a statement that only the shipping container was received (example: One cooler received from Company X in good condition. MMDDYY/ Initials).

11.15.3. Mention any discrepancies and allow corrections to be made by the client, sampler or courier. Also ask for any special instructions or important aspects of the project (i.e. rush turn-around times, impending hold-time violations, sample matrix specifics, etc.). Solicit the client's needs such as shipping containers, bottle orders, to speak with a project manager or any other requests. After the client's departure, complete any aspects of sections 5.1 through 5.15 left unfinished. Prioritize the new project within the existing workload and proceed with the highest priority.

11.16. After hours sample receipt instructions for non-sample administrative personnel (see Appendix 12)

12. CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Not applicable

14. POLLUTION PREVENTION

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2 All ice, or melted ice, that has been used to store or ship any soil samples, or in any

container with soil samples, must be allowed to melt through a 100 mesh screen, in order to comply with our USDA soil permit.

14.3 Ice chests that have been used to ship soil samples must be decontaminated with isopropyl alcohol during the cleaning process before being re-used.

15. WASTE STREAMS PRODUCED BY THE METHOD

15.1 The following waste streams are produced when this SOP is followed:

- Uncontaminated packing materials such as vermiculite, bubble wrap, foam inserts, plastic bags, paperwork, etc. These are collected in the uncontaminated lab trash cans, and are disposed of to the dumpster at the end of the day.

- Contaminated solid packing materials, including broken glass, caused by the breakage of sample containers during shipment. Dump the solid waste into a contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

- Contaminated melted ice and aqueous samples of unknown hazards, spilled when their sample container breaks during shipment. These materials are collected and disposed of in accordance with instructions from the Hazardous Waste Specialist, depending on the type of sample that was spilled.

- Contaminated melted ice and solid or soil samples of unknown hazards, spilled when their sample container breaks during shipment. These materials are collected and disposed of in accordance with instructions from the Hazardous Waste Specialist, depending on the type of sample that was spilled.

16. REFERENCES

16.1. Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth edition, January 2005, Appendix A.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Revision from previous version

17.1.1. Revised to include legal chain of custody

17.1.2. Reformatted.

17.2. Appendix 1 - STL Sacramento Sample Receiving Acceptance Policy

17.3. Appendix 2 - Sample Receipt Logbook

17.4. Appendix 3 - Lot Receipt Checklist

17.5. Appendix 4 – Short Hold Test Notification

17.6. Appendix 5 – Project Receipt Checklist

17.7. Appendix 6 – In House Chain of Custody

17.8. Appendix 7 – Lot Review Checklist #1

17.9. Appendix 8 – Lot Review Checklist #2

17.10. Appendix 9 – Flow chart

17.11. Appendix 10 – Chemical Warfare Degradates – Potential Hazards in Sample Receipt

17.12. Appendix 11 – Handling of Blood or Other Potential Infectious Materials

APPENDIX 1

STL SACRAMENTO
SAMPLE RECEIVING ACCEPTANCE POLICY

NELAC and STL Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. STL Sacramento will review your sample shipment against those requirements as listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

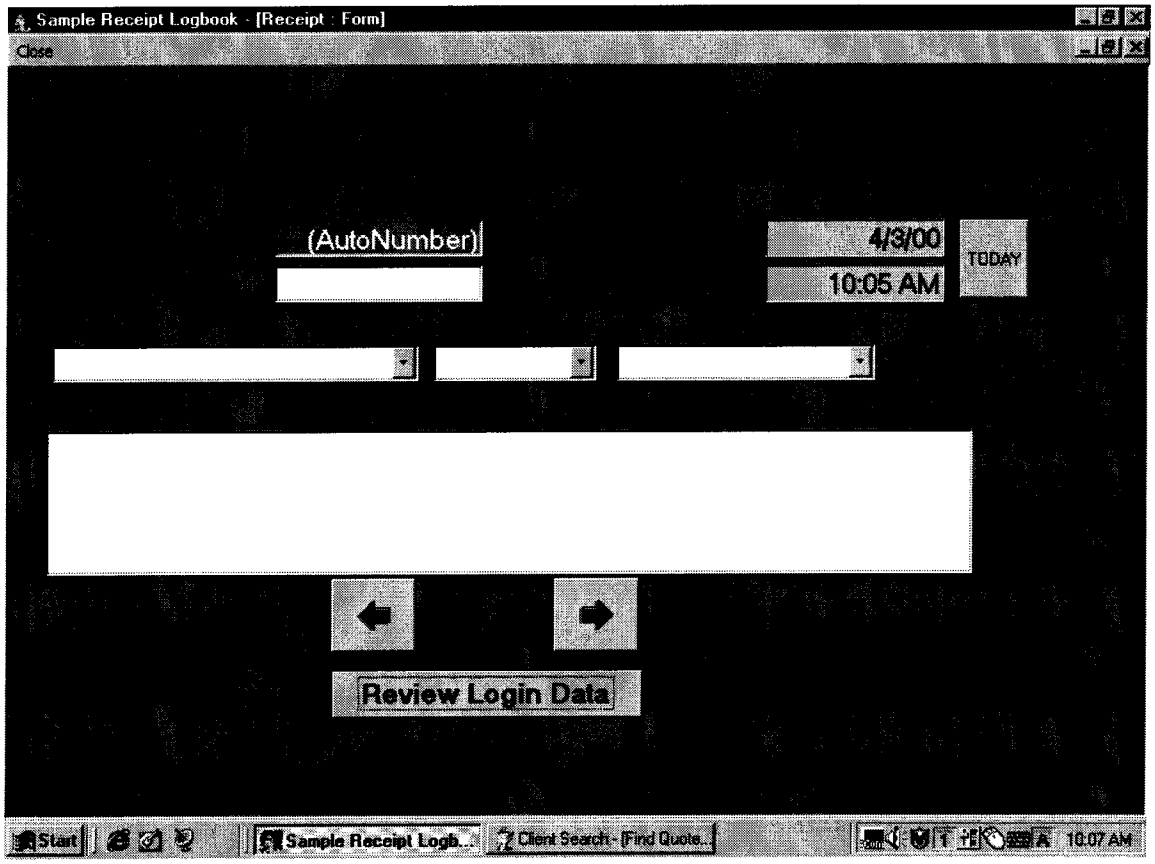
STL Sacramento requirements are as follows:

- ✓ Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples, shall be provided.
- ✓ Samples must be accompanied by written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law.
- ✓ Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- ✓ Drinking waters samples for Method 1613B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- ✓ The samples shall arrive at the laboratory with adequate remaining holding time for the analyses requested.
- ✓ Sufficient sample volume must be available to perform the requested analyses.
- ✓ Received samples must not exhibit obvious signs of damage, contamination or inadequate preservation.
- ✓ For samples undergoing chemical warfare degradate analysis, the sample must be screened for agent prior to shipment in accordance with appendix 10 of our Sample Receipt Procedure (SAC-QA-0003).
- ✓ Samples containing mammalian tissue will not be accepted without prior coordination with a project manager. Additional conditions for receipt and handling of tissue are outlined in appendix 11 of our Sample Receipt Procedure (SAC-QA-0003).

The laboratory will notify the client/Project Manager upon sample receipt if the samples fail to meet any of the above requirements.

When completing the chain of custody form, please do not forget to sign your name in the "relinquished by" box.

APPENDIX 2


Sample Receipt Logbook - [Receipt : Form]
Close
(AutoNumber)
4/3/00
10:05 AM
TODAY
Review Login Data
Start
Sample Receipt Logb...
Client Search - [Find Quote...
10:07 AM

APPENDIX 3

SEVERN TRENT **STL**

LOT RECEIPT CHECKLIST
STL Sacramento

CLIENT _____ PM_____ LOG # _____

LOT# (QUANTIMS ID) _____ QUOTE#_____ LOCATION_____

	Initials	Date
DATE RECEIVED _____ TIME RECEIVED _____	_____	_____
DELIVERED BY ☐ FEDEX ☐ CA OVERNIGHT ☐ CLIENT ☐ AIRBORNE ☐ GOLDENSTATE ☐ DHL ☐ UPS ☐ BAX GLOBAL ☐ GO-GETTERS ☐ STL COURIER ☐ COURIERS ON DEMAND ☐ OTHER	_____	_____
CUSTODY SEAL STATUS ☐ INTACT ☐ BROKEN ☐ N/A	_____	_____
CUSTODY SEAL #(S) _____		
SHIPPPING CONTAINER(S) ☐ STL ☐ CLIENT ☐ N/A	_____	_____
TEMPERTURE RECORD (IN °C) IR 1☐ 3 ☐ ☐ OTHER_____	_____	_____
COC #(S) _____	_____	_____
TEMPERATURE BLANK Observed:_____ Corrected:_____		
SAMPLE TEMPERATURE Observed:_____ _____ _____ Average:_____ Corrected Average:_____		
COLLECTOR'S NAME: ☐ Verified from COC ☐ Not on COC	_____	_____
pH MEASURED ☐ YES ☐ ANOMALY ☐ N/A	_____	_____
LABELED BY.....	_____	_____
LABELS CHECKED BY.....	_____	_____
PEER REVIEW _____ ☐ NA		
SHORT HOLD TEST NOTIFICATION SAMPLE RECEIVING	_____	_____
WETCHEM ☐ N/A	_____	_____
VOA-ENCORES ☐ N/A	_____	_____
☐ METALS NOTIFIED OF FILTER/PRESERVE VIA VERBAL & EMAIL ☐ N/A	_____	_____
☐ COMPLETE SHIPMENT RECEIVED IN GOOD CONDITION WITH APPROPRIATE TEMPERATURES, CONTAINERS, PRESERVATIVES ☐ N/A	_____	_____
☐ Clouseau ☐ TEMPERATURE EXCEEDED (2 °C – 6 °C)*1 ☐ N/A	_____	_____
☐ WET ICE ☐ BLUE ICE ☐ GEL PACK ☐ NO COOLING AGENTS USED ☐ PM NOTIFIED		

Notes:_____

*1 Acceptable temperature range for State of Wisconsin samples is ≤4°C.
LEAVE NO SPACES BLANK. USE "N/A" IF NOT APPLICABLE. INITIAL AND DATE ALL "N/A" ENTRIES.

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APPENDIX 4

STL Sacramento
SHORT HOLDING TEST NOTIFICATION

SEVERN TRENT STL

CLIENT _____ ☐ SHT ANALYSIS
LOT ID _____ ☐ PENDING HTV (ROUTINE TESTS)
DATE & TIME RECEIVED BY SAMPLE CONTROL _____ ☐ RUSH (24 - 48 - 72 HR TAT)
DATE & TIME RECEIVED IN GEN CHEM _____ ☐ SEE ATTACHED

CHEMIST NOTIFIED _____ QAS # _____

GENERAL ANALYSIS	SAMPLE #	ASSIGNED CHEMIST
☐ pH ☐ Alkalinity		
☐ $Cr6^+$ ☐ EC		
☐ Turbidity ☐ Color		
☐ SS ☐ SO_3		
☐ pH-Soils		
Lachat		
☐ NO_2+NO_3		
☐ NO_2		
☐ NO_3		
Method 365.3		
☐ OPO_4		
Method 300.0		
☐ NO_3 ☐ NO_2 ☐ OPO_4		
☐ Br ☐ Cl ☐ SO_4 ☐ F		
Metals		
☐ Dissolved Metals, Filter & Pres		
☐ OTHER		

Holding time already violated? ☐ Yes ☐ No

HTV filed? ☐ Yes ☐ No

☐ MS/MSD required on sample # ____ ☐ MS/DU required on sample # ___

Comments: _____

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APPENDIX 5 (BOTTLE LOT INVENTORY)

SEVERN TRENT STL

Bottle Lot Inventory

Lot ID:_____

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
VOA*																				
VOAh*																				
AGB																				
AGBs																				
250AGB																				
250AGBs																				
250AGBn																				
500AGB																				
____AGJ																				
500AGJ																				
250AGJ																				
125AGJ																				
____CGJ																				
500CGJ																				
250CGJ																				
125CGJ																				
PJ																				
PJn																				
500PJ																				
500PJn																				
500PJna																				
500PJzn/na																				
250PJ																				
250PJn																				
250PJna																				
250PJzn/na																				
Acetate Tube																				
_____"CT																				
Encore																				
Folder/filter																				
PUF																				
Petri/Filter																				
XAD Trap																				
Ziploc																				
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

h = hydrochloric acid s = sulfuric acid na = sodium hydroxide n = nitric acid zn = zinc acetate

Number of VOAs with air bubbles present / total number of VOA's

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APPENDIX 6

SEVERN TRENT STL

IN-HOUSE CHAIN OF CUSTODY

STL Sacramento

LOT # _____

LOCATION _____

SAMPLE CHECK OUT							SAMPLE CHECK IN						
SAMPLE (S)	CONTAINER	INIT	TEST	DATE	TIME	W	SAMPLE(S)	CONTAINER	INIT	DATE	TIME	Return Location	COMMENTS

1. Sample # (ex: 1,2,4-10)
2. Container (ex: AGB)
3. Init: your initials
4. Test: analysis sample is for
5. Date: date taken/returned
6. Time: time taken/returned
7. W: peer witness of checkout ("NA" unless legal COC)
8. Rtrn Loc: location samples returned to
9. Comments: (ie: empty, subsampled)

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APPENDIX 7

LOT REVIEW CHECKLIST #1

Lot # _____

SEVERN TRENT SERVICES

SHIPMENT RECEIPT:

____ COC/Letter of Transmittal signed and dated with time of receipt
____ Condition of samples noted on COC
____ Lot Receipt Checklist completed
____ COC generated for undocumented samples (from label information)

LOT FOLDER:

____ Means of delivery present on yellow sheet
____ Correct receipt date and time recorded
____ Sample ID(s) and collection date(s)/time(s) match COC or discrepancies noted on COC
____ Lot# labels are correct
____ Lot # entered in Excel spread sheet
____ Log # noted on yellow sheet

NOTIFICATION:

____ Anomaly sent via Clouseau
____ Wetchem notified of Short Holding tests
____ Metals notified of filtering and/or preservation
____ Rush E-Mail notification sent to PM and appropriate department(s)
____ HTV Filed for sample with expired hold times upon receipt (if applicable)
____ Subcontracted Samples

IN-HOUSE COC FOLDER:

____IN-HOUSE COC stapled to right side of folder
____ Bottle checklist stapled to the left side of folder
____ Folder filed

INITIAL/DATE _____

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APPENDIX 8

SEVERN TRENT SERVICES

LOT REVIEW
CHECKLIST #2
STL Sacramento

LOT # _____ DATE RECEIVED _____

SECTION I

LOG RELEASE

___ Verified Correct Quantims Quote
___ Verified Contact name/Report copy
___ Verified Billing name/address
___ Verified P.O. #
___ Verified Mail to address
___ Verified prices
___ Verified SAC's assigned properly
___ Verified sample Ids and date/time sampled
___ Client specific QC assigned
___ Lot Receipt Checklist complete
___ COC discrepancies resolved and documented
___ Verified TAT or due date
___ Confirmation sent to client
___ QAS assigned
___ Report Production Form completed
___ Log released
___ Verified Short Holding Time Notification

SUBCONTRACTED WORK

___ Confirmation of shipment (chain of custody of samples shipped)
___Expected Due Date entered in Target List system

SECTION I REVIEWED INITIAL/DATE _____

SECTION II

REPORT REVIEW

___ Signature page variables correct
___ Preliminaries documented
___ Table of contents correct
___ Correct TEQ Summary Sheet included
___ Data for all requested analyses
___ QA/QC data in control or anomalized
___ Case Narrative/Anomalies documented
___ EDD generated and completed

INVOICING

___ Additional costs added
___ Revenue Centers correct for additional items

SECTION II REVIEWED INITIAL/DATE _____

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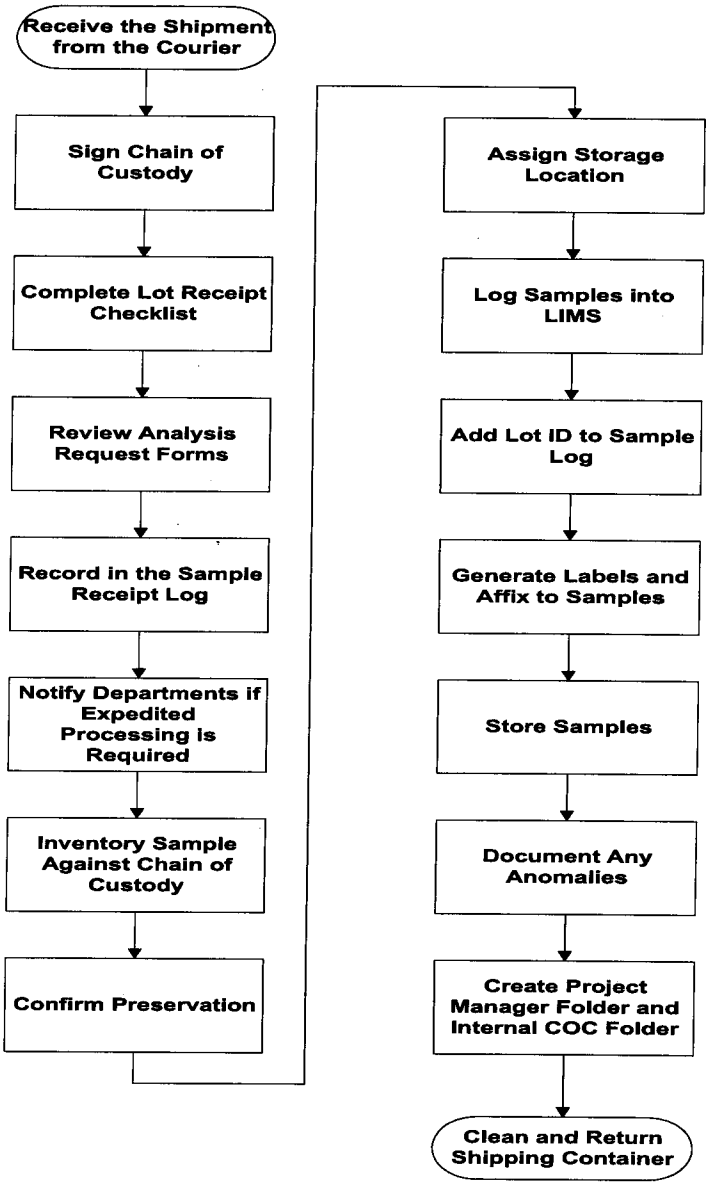
APPENDIX 9

Flow Diagram



APPENDIX 10

Chemical Warfare Degradates - Potential Hazards in Sample Receipt

Background

STL Sacramento regularly receives samples to be analyzed for degradates of chemical warfare agents. These degradates generally are no more toxic than most of the compounds we deal with every day. The fact that these compounds are degradates of chemical warfare agents does, however, present a different type of potential hazard for us. We have developed policies regarding the handling of such samples. The purpose of this document is to discuss these compounds and the potential hazards involved in handling them.

Please note that STL Sacramento does NOT analyze samples for actual chemical warfare agents such as mustard, lewisite, Sarin, GD, VX, phosgene and tear gas. Parent compounds are analyzed by laboratories that have specialized personnel training, security, and handling procedures.

The toxicity of the by-products of some chemical warfare materials are more than the parent compound. An example of this is VX and EA 2192. Testing protocol for degradates is not necessarily specific for the analyte being screened for and the scientific community has not come to consensus on what "positive" test results actually mean. For this reason, in all cases where positive screening data is received for the parent compounds, Corporate EH&S, the Project Manager, local EH&S staff and senior management must be consulted before deciding to accept and proceed with handling such samples.

Review of Agent Compounds

Chemical warfare agents fall into a wide variety of categories, ranging from relatively mild chemicals such as tear gas to lethal nerve agents such as Sarin. The two types of agents we are most concerned with are **nerve agents** and **blistering agents**. Other lethal agents have been developed and tested for use in chemical warfare; however, these other compounds are either extremely volatile and reactive (and therefore highly unlikely to be present in an environmental sample) or were never produced in significant quantities in the U.S.

Nerve agents

These are members of the organophosphate class of compounds. They are similar to many common household pesticides such as diazinon. The difference is that nerve agents are far more toxic to humans. The first nerve agent, Tabun, was discovered prior to World War I by a German pesticide company during the process of screening new compounds for use as pesticides. Once the toxicity of Tabun was determined, various governments began screening many related compounds. Out of this effort have come five established (non-classified, i.e. public domain) agents. --- **Tabun (or GA), Sarin (or GB), GD, GF, and VX.**

All of these compounds are toxic via inhalation, ingestion, skin contact, or just about any other route of entry into the body.

A **characteristic of these compounds is their volatility**. A more volatile nerve agent will disperse in air more effectively than a less volatile one.

Less volatile agents, on the other hand, will remain around on soil, vegetation, clothing, etc. and will therefore last longer. Out of these various agents, the only two produced in significant quantities in the U.S. are Sarin and VX. **Sarin is the most volatile** of the above agents while VX is the least volatile. Both are extremely toxic - a drop of pure VX barely visible to the naked eye is enough to kill a person through skin contact.

Blistering agents

These were the first of the modern agents developed specifically for military purposes. Unlike nerve agents, blistering agents are different mixtures of one compound, sulfur mustard, with other non-toxic chemicals which affect its dispersion characteristics.

The primary hazard with sulfur mustard involves skin contact. The term "blistering agents" is somewhat of a misnomer - it will kill you if you inhale enough of it, but this is not likely to occur as it is not particularly volatile under normal conditions. Significant quantities of sulfur mustard (a.k.a. HD or HT) have been produced in the U.S.

Chemical Agents in the Environment

It is unlikely (but never impossible) that we at STL Sacramento will receive a sample that contains a dangerous concentration of active agent. We **should not** receive samples from any areas known to be contaminated with active chemical agent (areas with buried drums, old munitions, etc.) because the Army policy for such sites is to destroy the agent onsite.

Additionally, most of the areas in which degradate analysis is required are areas in which agent was used decades ago - all active agent is likely to have degraded. Nonetheless, it is imperative that we know as much as possible regarding the compounds and the samples in order to protect ourselves against any potential hazard.

The behavior of these compounds in the environment has been extensively studied. Most of the information we have is from a study titled **Environmental Chemistry and Fate of Chemical Warfare Agents**. This study was prepared for the Army Corps of Engineers by Southwest Research Institute in 1994.

Sites containing chemical warfare related material or chemical warfare materials are divided into **"stockpile" and "non-stockpile" sites**. Stockpile sites are where the vast majority of CWM's are stored. Non-stockpile sites are where smaller amounts of CWM's are located. Non-stockpile sites may contain: buried CWM, chemical weapon production facilities, binary chemical weapons and miscellaneous CWM.

What is important to realize is that based on the **Survey and Analysis Report** prepared by the US Army Chemical Material Destruction Agency (11/93), there are "potential burials at 82 locations in 33 states, the US Virgin Islands and the District of Columbia.....Some of the 82 locations have multiple burial sites." Given this wide span of impacted areas, for every shipment received by STL Sacramento for CWM analysis, adherence to procedures listed in this document and the Corporate Safety Manual is strictly required.

Nerve Agents

Both of the compounds we are concerned with (Sarin and VX) undergo **hydrolysis in the presence of water**. This hydrolysis proceeds at different rates, depending upon the compound. At worst (cool temperature, normal pH, no dissolved ions, no microbes), either of these compounds getting into water would be degraded to extremely low levels ($< 1 \times 10^{-6}$ of the original concentration) within a couple of years. It is more likely that this level of degradation would occur much more quickly.

Degradation in soil is a far more complex issue. The rate of hydrolysis will depend upon a variety of factors, including soil moisture, pH, mineral content, microbes, temperature, etc. Most available studies show that these compounds last no more than a few days in the tested soil types.

Sulfur Mustard

In some ways, sulfur mustard behaves in a fashion similar to the nerve agents - i.e. it hydrolyzes rapidly in the presence of water. There is, however, an important difference. Under the right conditions, **the hydrolysis products of mustard can polymerize and form bubbles containing active mustard**. The mustard inside of these bubbles is shielded from further hydrolysis by the hydrolysis products. This has turned out to be a problem in areas where large amounts of mustard were dumped at sea. Fishermen in such areas have been injured when pulling up nets contaminated with blobs of active mustard. An indication that this may have occurred would be a biphasic sample. This situation can also occur in soil.

Potential Hazards to STL Sacramento Personnel

Following steps listed in this appendix and other safety policies will help reduce hazards to the greatest degree possible. However, such policies are no substitute for educated, observant personnel.

As always, you must think about what you are doing when you handle these materials. No policy can account for every potential situation. Staff are expected to follow all sample handling policies identified in the Corporate Safety Manual and steps following sample receipt listed in this appendix.

Soil and water samples will be screened for agent prior to their shipment to STL Sacramento, unless an exception has been granted by the Corporate Director of EH&S. Data should be reviewed at the project management and EH&S staff level to ensure samples are "safe" for handling. If the screening status is unknown (i.e. no data is available), project management personnel should be consulted. Samples will **not** be handled. If staff are unavailable, the cooler will be left in cold storage until the situation is resolved. The expiration of analytical holding times will not be considered as sufficient reason to handle/process CWM samples prior to receipt of screening data.

Positive "hits" on samples containing by-products of agents (like EA 2192) must also be reviewed at the project management and EH&S staff level to ensure samples are "safe" for staff to handle.

When in doubt, seek assistance from project management, operations manager, or EH&S staff. Corporate EH&S staff are also a resource which must be consulted before deciding to proceed with any "questionable" samples received.

Sample Receipt Procedures

⇒ Follow SAC-QA-003

⇒ Review screening data BEFORE opening the cooler if soil samples.

Note: Double gloves are required when handling chemical warfare degradate samples.

⇒ Following established safety policies, open the cooler, remove any paperwork, and check the interior condition.

⇒ All coolers from CWM sites will be initially opened in a fume hood. Once you have determined that there are no broken or leaking sample containers, the cooler may be moved to a bench top for further processing.

⇒ If a sample contains a broken or leaking sample, isolate the cooler in the hood and IMMEDIATELY contact the project manager, operation manager and/or EH&S staff.

⇒ Any sample which appears to be biphasic in appearance must be isolated. Immediate notification to project management, operation manager and/or EH&S staff is required.

⇒ When in doubt, get help regarding sample receipt. Worker health and safety is **paramount** to sample analysis.

⇒ Regardless of screening data, any cooler containing samples for degradate analysis (or any other samples from an area suspected of potential agent contamination) **should be inspected carefully** upon receipt. Anyone inspecting the samples, logging them in, or handling them for any other reason should observe all of STL Sacramento's regular safety procedures.

In addition, **two pairs of gloves** will be worn in order to minimize any potential for skin contact with toxic compounds. Please note that **skin contact appears to be the most likely potential route of exposure.** This is based on the fact that nerve agents are likely to have degraded leaving sulfur mustard as the most likely potential contaminant and due to the likelihood that the samples will be cold. The temperature in the cooler is important from the standpoint of safety as well as sample integrity - cold samples mean a significantly lower potential for any kind of toxic vapor formation. Coolers containing broken jars or bottles should be placed in a hood immediately and left there until the client has been contacted. EH&S staff and/or the project managers will give instructions regarding return to client or disposal based on the screening data.

In conclusion, it must be emphasized that these samples must be handled with the appropriate level of care. Observant, educated personnel are our best defense against exposure to any kind of toxic materials found in our samples.

APPENDIX 11
Handling of Blood or Other Potentially Infectious Material

Background

STL Sacramento has, upon occasion, received a variety of biological samples for various environmental analyses. Biological samples present a very different type of hazard than "typical" environmental samples. Depending on the type of sample delivered for analysis, and the types of analyses requested, a variety of additional precautions and protective measures may be required when receiving, processing and storing these samples.

Types of Biohazard Samples

There are many types of biohazard samples. Not all biological samples are necessarily biohazard samples. Some of the types of biohazard samples that have been received at STL Sacramento in the past include:

Human blood
Human tissue
Human breast milk
Rodent or other mammalian tissue
Human waste products, usually samples from municipal sewage treatment plants

While fish, crawfish, clams, plant tissue, grasses and such are all biological samples, they are not generally considered to be a biohazard threat.

Specific Hazards Associated with Biohazard Samples

The unique threat associated with biohazard samples is infectious diseases. Typically, these are Human Immuno Virus (HIV) and Hepatitis B. Other potential hazards include (but are not limited to) rabies, bubonic plague and the Hanta virus. Some of these hazards we are prepared to work with effectively, and others we are not.

Samples potentially infected with rabies, bubonic plague, or the Hanta virus require engineering controls that are not in place at STL Sacramento. Accordingly, we will not accept samples potentially infected by these diseases. These samples include whole rodents or other mammals, mammal parts, or homogenized mammal tissue. Mammal tissue samples that are known NOT to be infected with these diseases may be accepted for analysis under certain conditions. These samples must be homogenized and the sample tissue must be "fixed" in a 4% or higher formalin solution. The outside of the sample container must have been disinfected with a Centers for Disease Control (CDC) approved disinfectant after the sample was placed in the container but before it was shipped to us. Examples of this disinfectant solution are a 10% bleach solution or a 5% Lysol solution.

General Procedures

Universal precautions: All human and mammal blood, fluid and tissue samples are assumed to be infectious. All staff members will wear two pair of protective gloves when handling or working with biohazard samples. Safety glasses and a face shield are required. Fume hood sashes will be closed as far as possible, consistent with safe work practices. Lab coats will always be worn, buttoned up. Lab coats worn when handling biohazard samples will not be worn outside of the laboratory. When work is finished with biohazard samples, lab coats worn during the process will be sent out for cleaning. If they have been splashed or contaminated with any infectious sample, they will be disposed of as biohazardous waste. Workers will exercise caution to avoid injury with tools possibly infected from biohazard work, such as glass pipettes, metal spatulas, broken glass, etc. All waste material will be disposed of in appropriately marked containers as biohazardous waste. Workers with open wounds, sores or broken skin shall not handle biohazardous samples. Pregnant workers shall be especially familiar with and adhere to precautions to minimize the risk of transmission. Employees involved with handling human blood or tissue samples will be offered the opportunity to receive the Hepatitis

B vaccination series. This may be accepted, declined, or accepted at a later date.
Engineering and administrative controls: Signs will be posted on all doorways leading into areas where biohazardous samples are being handled. These signs will be clearly visible and will identify that biohazard work is in progress. When these signs are posted, personnel not involved in the work will stay out of the work area. If this is impractical, the biohazard work will be performed an isolated area that is clearly marked. No one may enter this area without permission from the sample administration technician or chemist doing the work. Personal protective equipment will be removed immediately upon leaving the area and disposed of or cleaned properly. Eating, drinking, use of tobacco products, gum, hard candy, applying cosmetics or lip balm and use of contact lenses are all prohibited in any areas where biohazard samples are being handled. Employees working with biohazard samples will thoroughly wash their hands with disinfectant soap when finished work and before leaving the lab. Work areas will be thoroughly cleaned and disinfected when biohazard work is complete. This includes properly disposing of bench paper and used equipment such as pipets, disinfecting all reusable equipment such as glassware, metal spatulas, and disinfecting work surfaces. Broken glassware that is potentially infected must not be picked up directly with the hands. Any trash cans or containers that may have been contaminated will inspected, cleaned and disinfected with an appropriate disinfecting solution.

Sample Receipt Procedures

Follow SAC-QA-0003
If advance notification is provided of incoming biohazard samples, contact EH&S, review this appendix and ensure that you are familiar with the safety procedures involved. Ensure that you have a clear workspace, that you know in advance where the samples will be stored, and that there is space available to store them.
Note: All potential biohazard samples must be kept in locked storage, either WR1 or WF1.
Note: Double gloves and a face shield are required when handling biohazard samples.

Biohazard samples shall be opened in a fume hood.
Following established procedures, open the cooler, remove any paperwork and check the interior condition.
If a shipping container has a broken or leaking sample, isolate the cooler in a fume hood and IMMEDIATELY contact the project manager and EH&S staff.
Note any comments or warnings on sample containers (including shipment paperwork) regarding specific threats or hazards.
When in doubt, get help regarding sample receipt. Your health and safety is of paramount importance.

The most likely methods of transmission of disease when handling biohazard samples are splashing infected blood or tissue onto an open cut or sore or into your eyes, mucous membranes or mouth. The likelihood of transmission via these routes can be almost completely eliminated by following proper procedures.

Exercise care when handling samples so that they do not drop or get knocked over.
Wear two pair of protective gloves – latex, vinyl or nitrile.
Don't work around biohazard samples with open cuts or sores.
Wear your safety glasses with a face shield.
Ensure that all skin is covered, such as your wrists and forearms
Wear your lab coat, properly fastened.

APPENDIX 12

After Hours Sample Receipt Procedures for Non-Sample Administrative Personnel

Normal business hours for receiving samples are Monday through Friday, 8:00 am to 6:00 pm, and Saturday, 8:00 am to 12:00 pm. In the event that samples are delivered outside of normal operating hours, and only non-Sample Administrative personnel are available to accept the delivery, the following procedures should be followed:

• If samples are delivered directly by a client, have him/her relinquish the COC, sign your name on the "Received By" line, note the time received, and make a copy of the signed COC for the client. Keep the original COC with the samples.

•If samples are delivered by a courier, and the COC is taped inside a cooler, note who delivered the samples and what time they arrived.

•Start a Lot Receipt Checklist (LRC), form QA-185, and complete the date/time received section and the custody seal section. Open the cooler/container and measure the temperature of the samples and/or temperature blank (if easily accessible) using the IR thermometer. Do not take the temperature of ice or packing material. Record the temperature(s) in the appropriate fields on the LRC. For clarity on which sections to complete, see the highlighted sections on the sample LRC posted next to these instructions.

•If samples are not contained in a cooler, document what kind of cooling agents were used, if any. Leave a note with this information with the COC.

•Place cooler/samples on a cart and store in the walk-in refrigerator.

•Send an email to "SACSC" to notify them that samples were received outside of normal hours, and include any pertinent information (i.e. when received, who delivered the samples, where they are located, sample receipt temperatures, etc.) to assist them in processing the samples when they return.

Title: Cleaning of Glassware (Organics)

Approvals (Signature/Date):

10/9/07	10/9/07
Alora Kuczynski — Date Technical Manager	Joe Schairer — Date Health & Safety Manager / Coordinator
PS 10/3/07 10/3/07	10/8/07
Pamela Schemmer — Date Quality Assurance Manager	Karla Buechler — Date Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This procedure is suitable for the cleaning of all general glassware for the Organic and Dioxin Prep departments.

1.2. Washing glassware requires attention to cleanliness to prevent artifacts and interferences during sample analysis.

2. SUMMARY OF METHOD

2.1. Organic contaminants are removed from extraction glassware by thermal destruction. After washing with a detergent solution and water, glassware is baked in a kiln for a prescribed period of time and temperature, allowed to cool to ambient temperature and then rinsed with an extraction solvent.

2.2. Dioxin contaminants are removed from the glassware by washing with a detergent solution and water, then rinsing with appropriate solvents. Soxhlet extraction glassware in the Dioxin prep department is to be pre-extracted with toluene for a minimum of 3 hours and not washed with a detergent solution.

3. DEFINITIONS

3.1. Glassware - Any item made of glass.

3.2. Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. It is the responsibility of the chemist and analyst to keep the workplace and glassware clean and neat. This requires full cooperation between each person in the laboratory.

4.2. Detergent solution residue can cause degradation of certain analytes. This problem is especially pronounced with glassware that may be difficult to rinse (e.g. 500 mL K-D flask, 300 mL round bottom flask, small test tubes). All items must be thoroughly rinsed with water to avoid this problem.

4.3. Glassware used for the analysis of Dioxin (PCDD/ PCDFs) samples is not kilned. High temperatures from the kiln and Dioxin residues can create PCB's that are an analyte of interest within the department. Kilning of glassware may also cause the formation of active sites on the glass surface that will irreversibly adsorb PCDD/PCDFs.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns or Requirements

5.1.1. Solvent rinsing glassware of any type with a squirt bottle is considered a high-risk activity. A face shield must be worn over safety glasses when performing this process.

5.1.2. Liquid detergent used in the laboratory is concentrated and caustic. Exercise caution when using it.

5.1.3. Before and after cleaning glassware, inspect it for cracks, chips, rub marks and sharp edges. If any glassware is found to be damaged and repairable, carefully clean it then put it aside for repair. If it cannot be repaired, discard it to the appropriate waste container. Do not use damaged glassware.

5.1.4. Be careful when working in tubs or sinks full of water and glassware. Do not "swish" hands around.

5.1.5. When washing glassware in a sink or tub, cut resistant gloves (Kevlar or MAPA heavy blue latex) must be worn, either under or over chemical protective gloves.

5.1.6. Use of a high temperature kiln to bake contamination out of glassware creates several hazards. All surfaces in the kiln, as well as the glassware, reach extremely high temperatures. After kilning, all glassware and the interior surfaces of the kiln must be allowed to cool before opening the kiln, reaching into it, or handling any of the glassware. Wear heat protective gloves when working with hot glassware, and exercise extra caution when working around the interior of a hot kiln.

5.1.7. Glassware **MUST** be allowed to cool before using flammable solvents on or around it.

5.1.8. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Only nitrile gloves should be worn when working with organic solvents, except for methanol. Latex gloves should be worn when using methanol.

5.1.9. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.10. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. 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.
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.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Despatch V Series kiln

7. REAGENTS

7.1. Detergent solution

7.2. Acetone

7.3. Methylene chloride

7.4. Hexane

7.5. Deionized Water

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. This section is not applicable.

9. QUALITY CONTROL

9.1. The department manager has the responsibility to ensure that this procedure is performed by an associate who has been properly trained and has the required expertise.

10. CALIBRATION

10.1. This section is not applicable.

11. PROCEDURE

11.1. Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

11.2. Glassware to be used for the extraction of organics compounds other than dioxins will be cleaned according to the following.

11.2.1. Inspect glassware for damage (e.g., star cracks). If damage is found carefully clean so that glassware may be repaired.

11.2.2. Glassware that has been in direct contact with a sample or with the sample extract must be rinsed thoroughly with acetone before washing.

11.2.3. If glassware is still visually oily, use other solvents, such as methylene chloride and/or hexane.

11.2.4. Allow glassware to dry in the hood before washing.

11.2.5. Wash glassware with appropriate amounts of detergent solution and water when, in the judgement of the chemist, it is necessary to remove residue adhering to the glassware.

11.2.6. Rinse all glassware well with tap water, and place in the kiln (see Section 6.4).

11.2.7. **Do not place graduated cylinders or volumetric glassware in a kiln.** The measurement volume may change due to warping. Allow to drain well and air dry. Be sure the glassware drains properly to avoid puddles inside the glassware. Put clean, dry, volumetric glassware away.

11.2.8. Glassware that is not to be kilned, either due to volumetric usage or time constraints, may be washed with detergent solution and rinsed with water, then rinsed three times each with acetone, then hexane, and finally methylene chloride.

11.3. Glassware used for the extraction of dioxins will be cleaned according to the following procedures.

11.3.1. Glassware should be rinsed with acetone and washed with a detergent solution and water as soon as practical after use. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

11.3.2. After detergent washing, glassware should be rinsed thoroughly with water, then acetone (until all water is removed), then toluene (all water should be removed first – if a white emulsion forms, repeat acetone rinse until all water has been removed), then hexane, and last methylene chloride.

11.3.3. Do not kiln reusable glassware in an oven as a routine part of cleaning.

Kilning may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated kilning of glassware may cause the formation of active sites on the glass surface that will irreversibly adsorb PCDDs/ PCDFs.

11.3.4. Do not wash Soxhlet extraction glassware with detergent solution. Soxhlet extraction glassware should be pre-extracted with toluene for a minimum of 3 hours.

11.3.5. If the glassware will not be used immediately, allow to dry and store inverted.

11.4. Kilning procedure

11.4.1. Glassware to be kilned must be cleaned according to Section 6.2.

11.4.2. Ensure that the kiln is turned on and the appropriate temperature program is entered.

11.4.3. Carefully stack the glassware in the kiln.

11.4.4. Use restraining bars to prevent glassware from rolling out of the kiln when the doors are opened.

11.4.5. Kiln glassware for the amount of time specified by the MIC6000 Temperature Controller box on the oven. Profile 1 is programmed with a 3-segment temperature profile:

11.4.5.1. Thirty minutes to ramp to 752°F, hold for 1 hour at 752°F.

11.4.5.2. One hour to ramp down to 450°F, hold 5 minutes.

11.4.5.3. One hour and fifteen minutes to ramp down from 450°F to 100°F, heat coils are off.

11.4.6. Do not open the kiln until the temperature is below 100°F. A heat wave from the kiln can cause burns or eye damage.

11.4.7. Wearing heat resistant gloves, remove the glassware from the kiln and place it back in its original storage container.

11.4.8. For more information regarding the operation and maintenance of the kiln, see SOP# SAC-OP-4177.

Warning: Do not handle the glassware until it has cooled! Do not attempt to solvent rinse the glassware until it has completely cooled!

12. CALCULATIONS/DATA REDUCTION

12.1. This section is not applicable.

13. METHOD PERFORMANCE

13.1. The department manager has the responsibility to ensure that this procedure is performed by an associate who has been properly trained and has the required expertise.

14. POLLUTION PREVENTION

All waste will be disposed of in accordance with Federal, State and Local regulations.

14.1. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

14.2. Transfer waste from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

14.3. The use of a high temperature kiln may reduce the volume of organic solvent used in glassware cleaning, and the amount of waste solvent that is produced. Thoroughly evaluate what type of cleaning is necessary depending on the type of glassware being used, the type of contamination present on the dirty glassware, and the types of analyses the glassware will be used for in order to determine the appropriate cleaning procedure that minimizes resource use and waste production.

15. WASTE MANAGEMENT

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

15.1. Waste flammable solvent from rinsing selected glassware is collected in one-liter to four-liter jars or jugs at the bench during use. When full, at the end of the day or at the end of the shift, this is poured into a 55-gallon steel flammable waste drum kept in the H-3 closet. When full or after no more than 75 days, this drum is moved to the waste collection area for shipment.

15.2. Waste methylene chloride from rinsing selected glassware is collected in one-liter to four-liter jars or jugs at the bench during use. When full, at the end of the day or at the end of the shift, this is poured into a 55-gallon steel waste methylene chloridedrum kept in the H-3 closet. When full or after no more than 75 days, this drum is moved to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. This section is not applicable.

17. METHOD MODIFICATIONS

17.1. This section is not applicable.

18. ATTACHMENTS

18.1. No attachments are present.

19. REVISION HISTORY

19.1. WS-OP-0011, Revision 3, Effective 10/9/07

19.1.1. The SOP format was updated to TestAmerica format.

19.1.2. Equipment list was added.

19.2. SAC-OP-0011, Revision 2.1, Effective 8/29/05

19.2.1. Updated safety portion of the SOP.

19.3. SAC-OP-0011, Revision 2, Effective 8/12/03

19.3.1. SOP was updated to include glassware cleaning for Dioxins.

19.4. SAC-OP-0011, Revision 1, Effective 7/17/01

19.4.1. Updated to reflect change in ownership from Quanterra to STL.

19.5. SAC-OP-0011, Revision 0, Effective 3/5/98

Reviewed 10/08/2004
Reviewed 3/14/2006
Reviewed 2/28/2007
Reviewed 2/22/2008

Title: Nonconformance Corrective Action System

Approvals (Signature/Date):

Lisa Stafford — 3/31/08 — Date Technical Manager	Joe Schairer — 3/31/08 — Date Health & Safety Manager / Coordinator
Pamela Schemmer — 3/31/08 — Date Quality Assurance Manager	Karla Buechler — 3/31/08 — Date Laboratory Director

This SOP was previously identified as SOP No. SAC-QA-0023.

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1. PURPOSE

1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The STL Quality Assurance Management Plan (QMP) and STL Sacramento Laboratory Quality Manual (LQM) require documentation of instances of deviations from established control limits, approved standard operating procedure (SOPs), or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from STL Sacramento policies and procedures and documented client specifications including root causes and corrective actions taken to remedy the nonconformance. The NCM will be stored in electronic form in a database called Clouseau, with any supporting material filed in QA as necessary.

1.2. This document applies to procedures, services, analytical data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. The system used to handle the sample receiving and client-related issues, including holding time violations (HTVs), emphasizes the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.

1.3. Nonconformances can be identified by associates in the course of their daily operations or by external parties (i.e., customers and representatives of customers) through reviews of records, audit, or proficiency testing.

2. RESPONSIBILITIES

2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, which might affect the quality of STL Sacramento's product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.

2.2. **Department Manager (DM):** Each department manager is responsible for the review of NCMs within 24 hours of initiation to ensure that problems that might affect quality are adequately described and that personnel are assigned to correct them. Department managers review hardcopy or electronic versions of NCMs and forward them to the appropriate project manager. Together with project managers and Quality Assurance personnel, department managers are responsible for determining the appropriateness of planned corrective actions.

2.3. **Project Manager (PM):** The project manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The project manager communicates conformance problems to clients and documents decisions made with clients. The project manager

ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that repreparation and analysis of a sample was done. The project manager may withhold final reports to clients until corrective actions agreed to with the client have been completed. Project managers are also responsible for initiating an NCM for client complaints or inquiries. The PM is responsible for reviewing the NCM within 24 hours of receipt.

2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for reviewing all initiated NCMs within 72 hours (24 hours of receipt) to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and spot audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.

2.5. **Operations Manager**: The operations manager shall ensure that corrective actions are correct and have been implemented, and that NCMs are written in a concise, clear manner. The operations manager review and concurrence shall be documented in the database as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.

2.6. **Laboratory Manager:** The laboratory manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.

2.7. **Corporate QA Director**: The STL Quality Assurance Director should be notified of any continuing nonconformances that are not properly addressed by operations or where the root cause cannot be identified.

3. SAFETY

3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2. Procedures shall be carried out in a manner that protects the health and safety of all associates.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported **immediately** to a laboratory supervisor.

4. PROCEDURE

4.1. When to Initiate a Nonconformance Memo

4.1.1. Lab associates are to initiate an electronic nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or standard materials deviate from established specifications. An NCM may also be initiated to document and observation noted during the course of the analysis that may or may not have an effect on the resulting data. Such an observation would include unusual color or odor. All nonconformances with the exception of matrix-related failures require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions). ALL holding time violations (HTVs) require an NCM.

4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, bench sheets, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPPs, Quality Assurance Summaries, or equivalent systems). Any intentional deviation from an SOP must be pre-approved by the QA manager and Supervisor. Any deviation from an SOP or client requirement not previously approved must be documented using the NCM process.

4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple lot numbers and samples, but normally a separate NCM would be initiated for different nonconformance issues. If the nonconformance involves projects for multiple project managers, then the NCM will need to be routed to each project manager.

4.1.3.1. An NCM may be initiated to document client complaints. See Policy QA-013-SAC.

4.2. How to Process the NCM using Clouseau

4.2.1. Initiating the NCM

4.2.1.1. While properly logged into a PC where Clouseau has been installed, start the Clouseau program.

4.2.1.2. At the Main Menu, select New.

4.2.1.3. Enter the information required by the wizard. At a minimum select the Production Area, Category (anomaly, deficiency or observation. (Note: observations are not reviewed by department managers or QA)), NCM Type, and NCM Description.

4.2.1.4. Select whether the nonconformance affects entire lots, entire batch, specific sample, specific analysis (work order) or other. "Other" should only be used in cases such as client complaints or instrument tag outs, which do not affect specific samples. If using one of the "entire" options, delete extra samples by highlighting and using the

delete key on the keyboard. The view may be resorted by clicking on one of the headers.

4.2.1.5. Complete the Comments/Details and Corrective Action sections of the Create NCM Form. These sections may be filled out jointly with the department manager. Consult the project manager or the operations manager and QA manager if the department manager and associate are uncertain of corrective actions. Be objective and specific but brief. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).

4.2.1.5.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples.

4.2.1.5.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.2.1.6. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff before the NCM is considered closed.

4.2.1.7. Save the NCM using the appropriate command in Clouseau. When presented with the Send E-Mail form, the department manager of the affected Production Area will be listed in the "Notify Now" box, while affected Project Managers and QA staff will be listed in the "Notify Later" box. Verify that these names are correct. If any personnel should be informed that are not listed, add their names to the "Notify Now" box by double-clicking on their names in the Personnel box.

4.2.1.8. Initiate the NCM notification process by selecting SEND EMAIL.

4.2.2. Department Manager Review and Approval

4.2.2.1. The department manager will receive notification of the NCM via e-mail. The department manager must log into a computer workstation where Clouseau has been installed, and run Clouseau.

4.2.2.2. Using the Review NCM form, select the NCM to be reviewed and highlight.

4.2.2.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. If the corrective action has not been determined, the situation must be referred to the project manager and the operations manager for

resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations manager needs to be made aware of the issues, the QA staff will notify the operations manager using the Under Review/Send Email options of Clouseau.

4.2.2.4. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired. Refer to Section 4.3.

4.2.2.5. The department manager will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the department manager. This is Operations' commitment to rectify the problem. The department manager selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process, which will now route the NCM to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager ***must*** be notified ***immediately***.

4.2.3. Project Manager Review, Client Notification, and Project Documentation

4.2.3.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate using the Client Notification Form in Clouseau the date and method of notification and client's response.

4.2.3.2. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. Notify the department manager of any changes made to the corrective action plan.

4.2.3.3. The project manager must select the "PM" button and document how client notification was done, and what, if any, response was made. If multiple projects are associated with one NCM, each project must be highlighted in this screen, and appropriate notification documented.

4.2.3.4. The project manager selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing to the QA office. The QA office then must review the NCM in a timely manner. This approval and routing should be done within 72 hours of initiation, or 24 hours of receipt by the project manager.

4.2.3.5. If the nonconformance involves analytical work in process, the **final** report cannot be released until a project manager has approved the NCM. If it is found that erroneous analytical data (e.g., from data

validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned into the QA staff if corrections are needed in the database (QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.2.4. Quality Assurance Review and Trending

4.2.4.1. The QA staff shall review all NCMs for conformance with standard laboratory practices and policies.

4.2.4.2. NCMs will be reviewed to ensure that the corrective action was completed and effective in addressing the root cause of the nonconformity to prevent recurrence.

4.2.4.3. Clouseau's reporting and tracking system will be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.2.4.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic problems must be documented by the responsible laboratory area along with supporting evidence.

4.2.4.5. The QA staff shall conduct spot follow-up assessments to confirm that correction of systematic problems is successful. These assessments are done on at least an annual basis.

4.2.4.6. The approval of the QA manager or designee is required in Clouseau to indicate that the NCM has been closed.

4.2.4.7. The QA office maintains a central file/record of all closed NCMs. For systematic or data recall issues, supporting material may also be retained in the QA office.

4.3. Instrument/Equipment Nonconformance Tag

4.3.1. Instruments and equipment that habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a clearly visible tag or sign indicating the nonconforming condition.

4.3.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM. Identify the instrument by name and/or identification number and briefly describe the problem.

4.3.3. Upon saving the NCM, the NCM number indicated by Clouseau shall be recorded in the instrument's maintenance log.

4.3.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record the back to control information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

5. DEFINITIONS

5.1. ***Nonconformance:*** an unplanned deviation from an established protocol or plan. The deviation may be the result of STL Sacramento's actions, then termed a **deficiency**, or the result of events beyond the control of STL Sacramento, then termed an **anomaly**. An **observation** is phenomena that may need to be included in the case narrative.

A nonconformance exists when:

5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrates a **systematic** deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed to matrix effects must be documented, but an NCM is not required. A procedure is not performed as described in the applicable SOP or QA Policy, ***except*** in cases where the procedure has been performed according to a client-specified document STL Sacramento has agreed to follow (e.g., EPA SOWs and QAPPs).

5.1.2. A practice or procedure is not performed as described according to a client or project document that STL Sacramento has agreed to follow.

5.1.3. Purchased materials or services are determined to be defective and their use would affect data quality.

5.1.4. ANY holding time violations (HTVs) occur regardless of what or whose actions caused them including ACTS of GOD.

5.1.5. Any lab communication (such as description of sample or sample performance) from the analyst to department manager, PM, or QA.

5.1.6. A formal NCM is not required for routine minor instrument maintenance, malfunctions, and power failures which can be documented in instrument maintenance logbooks.

5.2. ***Corrective action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.

5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.

5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Reference Documents

6.1.1. STL Sacramento Laboratory Quality Manual (LQM), current revision.

6.1.2. STL Quality Management Plan (QMP), current revision

6.1.3. ANSI/ASME NQA-1, Chapter II, Basic Requirement 15 "Control of Nonconforming Items." Supplement 15S-1 "Supplementary Requirements for the Control of Nonconforming Items."

6.1.4. ANSI/ASQC Q94-1987, "Quality Management and Quality System Elements - Guidelines," Section 14.0 "Nonconformity" and Section 15.0 "Corrective Action."

6.1.5. Clouseau Program Documentation.

6.2. Appendices

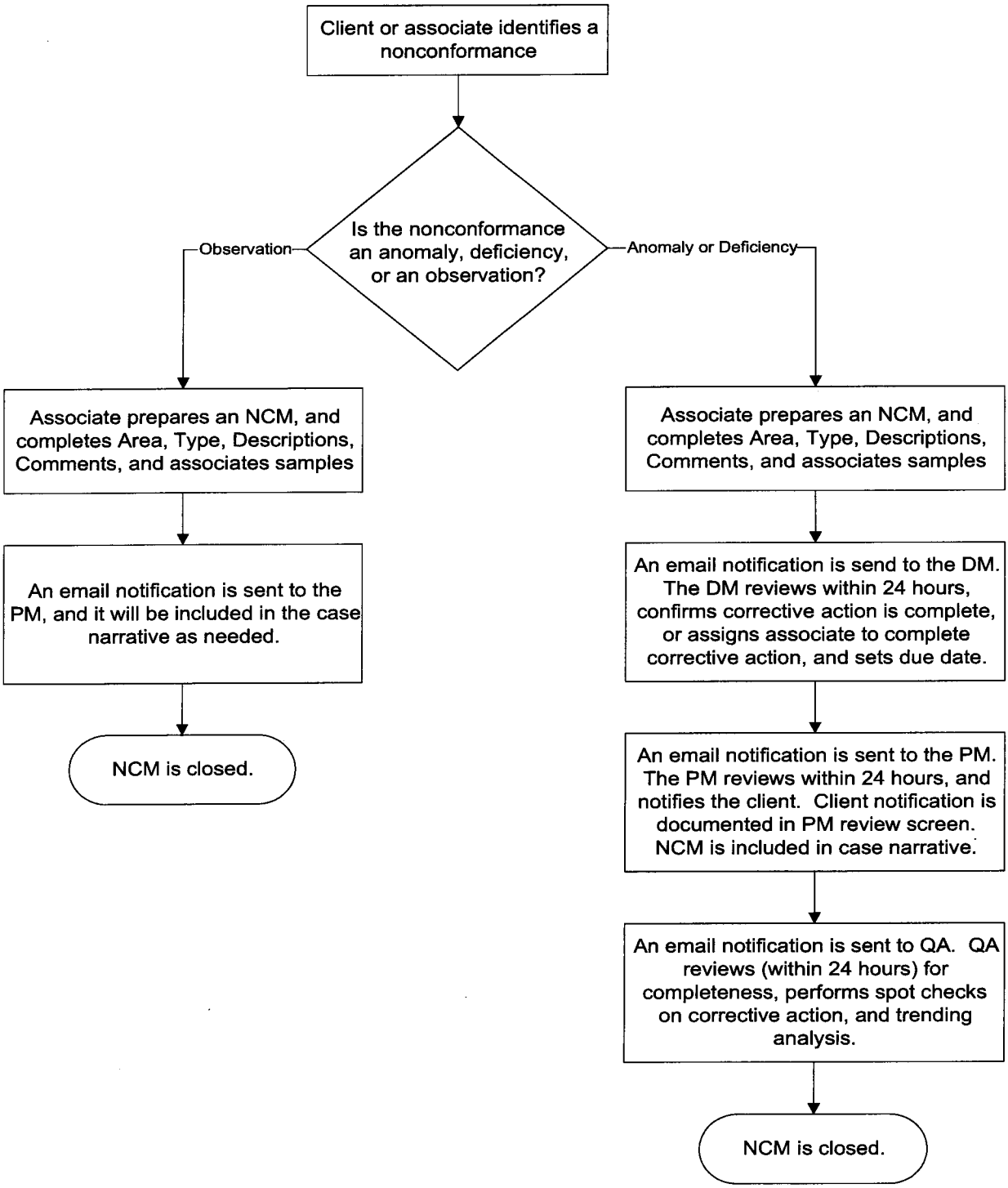
6.2.1. Attachment A: Nonconformance Memo Generated from Clouseau

6.3. Changes from previous version

6.3.1. Miscellaneous typographical errors were fixed.

6.3.2. New example NCM with latest logo inserted.

6.3.3. Flow Chart for Internal NCM



ATTACHMENT A

EXAMPLE LABORATORY NONCONFORMANCE MEMO (GENERATED BY CLOUSEAU)

Clouseau Nonconformance Memo

SEVERN TRENT STL

NCM #: **07-29880**
NCM Initiated By: Doug Weir
Date Opened: 05/16/2003
Date Closed: 05/19/2003

Classification: **Deficiency**
Status: **CLOSED**
Production Area: Dioxin Data Review
Tests: 8290
Lot #'s (Sample #'s): G3E050130 (13,14,15), G3E070000 (571),
QC Batches: 3127571,

Nonconformance: Laboratory Contamination
Subcategory: Laboratory contamination affecting blank or samples

Problem Description / Root Cause

Name	Date	Description
Doug Weir	05/16/2003	Samples G3E050130-13,14,15,14MS and 14MSD have been "B-flagged" because the method blank has OCDD contamination above the detection limit. OCDD is a ubiquitous laboratory contaminant. As per our SOP (Section 9.1.4.1), the data is deemed acceptable if the OCDD concentration is <5x the specified reporting limit, as it is in this case, and the sample is given a "B" qualifier. There is no adverse impact from this anomaly, and no corrective action is necessary.

Corrective Action

Name	Date	Corrective Action
Doug Weir	05/16/2003	

Client Notification Summary

Client	Project Manager	Notified	Response	How Notified	Note
[redacted]	WEIDENFELDR	05/19/2003	05/19/2003	by narrative	

Response	Response Note
No response saved	

Quality Assurance Verification

Verified By	Due Date	Status	Notes
STAFFORDL		Verified/completed	

Approval History

Date Approved	Approved By	Position

Date Printed: 5/19/03

Page 1 of 1

Title: Quality Control Program

Approvals (Signature/Date):

Pamela Schemmer 2/21/08	Karla Buechler 2/21/08
Pamela Schemmer — Date	Karla Buechler — Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. QA-003-SAC.

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OBJECTIVE:

This policy describes the STL Sacramento program of routine analytical quality control (QC) activities. The objective is to generate QC data that demonstrate that the analytical process is in control and that the data meet client and method requirements.

SCOPE:

This policy is to be enforced and followed throughout the laboratory.

POLICY:

1. Assessments of QC data relative to control limits determine the acceptability of sample test results. Whenever control criteria are not met, the data must be evaluated to determine appropriate corrective action. The initial evaluation is made by the analyst, frequently in conjunction with data review software and/or senior analysts or supervisors. Further technical evaluation of the data or data review software output is conducted by second-party data reviewers. Corrective action decisions, particularly whether or not to reanalyze samples, should be done in consultation with the client to the extent possible when operating under project-specific QA plans. Requirements for assessment and corrective action are described in the attachments to this policy. Details concerning technical data review and documentation of the reviews are described in "Technical Data Review Requirements" (Policy QA-012-SAC).

2. Application of STL Sacramento's standard QC program should be communicated to the client prior to acceptance of work. At the same time, every effort must be made to understand clients' special project requirements. Generally, laboratory project managers serve as a liaison between the clients and the laboratory staff to ensure that requirements are properly communicated in writing to both parties. In the event that alternative QC procedures are not specified by our clients, these standard QC protocols must be followed to ensure the generation of legally and scientifically defensible analytical data.

3. Successful implementation of this QC program requires that it is clearly understood by all STL Sacramento staff. Training based on this policy will be conducted at the laboratory and provided to new personnel as appropriate for their functions.

4. STL Sacramento's QC program applies to the following regulatory programs:

   4.1 RCRA and SW-846 Methods - All routine analytical projects performed using SW-846 methods must comply with the requirements described in STL Sacramento Laboratory Quality Manual (LQM), and Attachment I to this policy. The Quality Control sections of analytical standard operating procedures (SOPs) referencing SW-846 methods must be consistent with the requirements in Attachment I.

   4.2 CWA and 40 CFR Part 136 Listed Methods - Any analytical work conducted in support of an NPDES permit or other Clean Water Act compliance activities, must meet the quality control specifications shown in STL Sacramento's LQM. The quality control requirements for the specific methods listed in the LQM define the minimum requirements that must be given in the laboratory analytical SOPs.

   4.3 Safe Drinking Water Projects – Any analytical work conducted in support of SDWA compliance activities must meet the quality control specifications shown in STL Sacramento's LQM. The quality control requirements for the specific methods listed in the

LQM define the minimum requirements that must be given in the laboratory analytical SOPs.

4.4 Other programs or projects with clearly defined QC requirements

4.4.1. The differences between STL Sacramento's standard QC program and special project requirements must be specified in project documents. These documents may include Quality Assurance Project Plans (QAPjPs), Quality Assurance Program Plans (QAPPs), Sampling and Analysis Plans (SAPs), Statements of Work (SOWs), project-specific Quality Assurance Summaries (QASs), SOPs, contracts, or other approved documents.

4.4.2. Documents describing special project requirements must be reviewed and approved by appropriate QA and operations staff.

4.4.3. If the special project requirements appear to result in modifications that contradict federal or state regulatory requirements, the variance must be noted in writing and communicated to the client. A record of this communication must be retained as a permanent part of the project file.

4.4.4. Any special client's project requirements must be communicated to STL Sacramento's analysts in advance of releasing samples for analysis, and the work must be clearly differentiated in the analytical documentation, otherwise Attachment I requirements will be followed.

4.5 Projects without specific QC requirements

Any projects, for which no specific QC program is specified, regardless of the source of the analytical methods being used, must follow the requirements shown in Attachment I.

5. Changes from Previous Revision

5.1 The process for generating control limits has been updated to reflect current practices.

5.2 Language regarding Marginal Exceedences has been added.

ATTACHMENT I
STL SACRAMENTO QUALITY CONTROL PROGRAM

INTRODUCTION

This quality control (QC) program is based on the requirements in "Test Methods for Evaluating Solid Waste", USEPA SW-846, Third Edition with promulgated updates. It applies whenever SW-846 analytical methods are used. It also applies in whole or in part whenever project requirements fail to specify some aspect of QC practices described here. It does not apply when other well defined QC programs (e.g. CLP, CLP-like, DOD, AFCEE, USACE, or NFESC) are specified. This policy represents STL Sacramento's base QC program for environmental analyses.

Details concerning instrument calibrations, tunes, and QC that are required for specific methods (e.g., interference check samples for ICP and isotopic spikes for dioxin procedures) are not given here. Refer to the method standard operating procedures (SOPs) for information about the frequency, assessment and corrective action required for additional QC elements.

1. DEFINITIONS

1.1 QC Batch — The QC batch is a set of up to 20 field samples plus associated laboratory QC samples that are similar in composition (matrix) and that are processed within the same time period using the same reagent and standard lots.

1.2 Surrogates — Surrogates are organic compounds similar in chemical behavior to the target analytes, but that are not normally found in environmental samples. When utilized, surrogates are added to all samples in a batch to monitor the effects of both the matrix and the analytical process on accuracy.

1.3 Method Blank — An interferent-free blank matrix similar to the sample matrix being tested. This analytical control is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination for a given procedure on a given matrix. Examples of method blank matrices are a volume of deionized or distilled laboratory water for water samples, a purified solid matrix for soil/sediment samples, or a generated zero air.

1.4 Instrument/Calibration Blank — The instrument blank is prepared using the same solvents and reagents (e.g. hexane, methylene chloride, or reagent water) used to dilute the prepared sample extracts or digests. Unlike the method blank, it is analyzed without being subject to the preparation steps of the analytical procedure. It is used to monitor laboratory or reagent contamination introduced at the instrumental analysis phase of work. For procedures without a separate preparation step, an instrument blank is equivalent to the method blank, and serves the same purpose.

1.5 Laboratory Control Sample — A laboratory control sample (LCS) is prepared using a well characterized matrix (e.g. reagent water or Ottawa sand) that is spiked, with known amounts of representative analytes. Alternate matrices (e.g. sodium sulfate) may be used for soil analyses when Ottawa sand is not appropriate. As part of a QC batch, it accompanies the samples through all steps of the analytical process. The LCS is used to monitor the accuracy of the analytical process independent of possible interference effects due to sample matrix.

1.6 Duplicate Laboratory Control Sample — Duplicate laboratory control samples (LCS/LCSD, also called a DCS pair) consist of a pair of LCSs analyzed within the same QC batch to monitor precision and accuracy independent of sample matrix effects. This QC sample is prepared at the request of the client and/or may be prepared when insufficient sample volume is received to prepare and analyze an MS/MSD pair and precision information within a preparation batch is required. The LCS/LCSD is intended to provide information regarding the precision of the measurement process within a preparation batch. Precision may also be performed using LCS between subsequent preparation batches.

1.7 Matrix Spike and Matrix Spike Duplicate

1.7.1. Matrix Spike — A matrix spike (MS) is a replicate portion of one field sample in the QC batch that is spiked with known amounts of target analytes. An MS is spiked with the same analytes at the same concentrations that are added to the LCS. As part of the QC batch, it accompanies the field samples through all steps of the analytical process.

1.7.2. Matrix Spike Duplicate — A matrix spike duplicate (MSD) consists of an additional portion of the same sample used to prepare the MS. This portion is spiked and processed exactly as the MS.

1.7.3. The MS and MSD results are used to determine the effect of the sample matrix on the precision and accuracy of results. Due to the potential variability of the matrix of each sample, the MS and MSD results may not have immediate bearing on any samples except the one spiked.

1.8 Sample Duplicate — A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

1.9 Internal Standard — An internal standard (IS) is a compound or element with similar chemical characteristics and behavior in the analysis process to the target analytes, but is not normally found in environmental samples. The internal standard is usually added after sample preparation. The primary function of the internal standard is quantitation; however, it also provides a short-term indication of instrument performance.

For isotope dilution methods, internal standards are added during sample preparation and are used for quantitation. The effect of matrix effects on method performance may be evaluated via the isotopically labelled compounds used as internal standards. These isotopically labelled compounds are analogs of target analytes and are spiked into each sample at the time of extraction. Therefore, matrix effects on method performance can be

judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample.

1.10 Control limits — The reported control limits are either based on laboratory historical data, method requirements, or project data quality objectives. The control limits represent the estimated uncertainty of the laboratory process.

2. BATCH QC ELEMENTS & BATCH PROCESSING

2.1 A QC batch is designed to determine the quality of the analytical results obtained for a group of up to 20 field samples in terms of accuracy and precision. With some exceptions as described in Sections 3.6 through 3.8 below, the minimum QC elements for each QC batch are

- one method blank (MB),
- one laboratory control sample (LCS),
- one matrix spike (MS), and
- one matrix spike duplicate (MSD).

2.2 The LCS and MS/MSD are spiked at the same concentrations and with the same analytes. The spike analytes should encompass all analytes in the routine daily calibration standards. Additional, "non-routine" analytes may be requested to be spiked on a project specific basis.

2.2.1. All analytes encompassed by a given analysis must be spiked in at least two LCS over a two year period.

2.3 The identity of each QC batch must be documented and traceable, e.g., each batch of field samples must be clearly associated with the applicable QC samples.

2.4 To the extent possible, samples that require a preparation step should be analyzed together with their associated QC samples. If the samples in a given QC batch require separate analytical runs, the minimum batch QC in each run is an acceptable MB or instrument/calibration blank. To the extent possible, the QC samples should not be analyzed independent of the field samples on a different instrument. QC samples are not to be preferentially run on one instrument over another instrument.

2.5 For analytical procedures that do not include a separate extraction or digestion (e.g., volatile organic analysis by purge and trap), the QC batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event and analytical batch. That is, the same tune period, calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

2.6 Field QC samples (e.g., trip blanks, equipment rinsates, and field duplicates) count as individual samples, therefore, they add to the QC batch count. Samples that require simple reanalysis (e.g., dilutions to adjust a sample extract to the working range of the instrument), as opposed to re-extraction or re-digestion and reanalysis, do not count as additional samples in the QC batch.

2.7 MS/MSD pairs are performed to demonstrate matrix effects and precision within a preparation batch. They are not the only acceptable means of demonstrating precision.

2.7.1. As requested by clients or required by some methods, batch precision may also be demonstrated through the analysis of sample duplicates (DUs). However, the client

should be advised that a DU is less likely to provide usable precision statistics depending on the likelihood of finding concentrations below reporting limits.

2.7.2. A duplicate LCS (DCS) may be used to demonstrate method batch precision independent of the client's matrix when the client has not supplied sufficient sample quantity to prepare an MS, MSD or DU, or as a program requirement, e.g. Arizona.

2.7.3. On-going monitoring of LCS results can be used to determine long-term (a.k.a., between batch) precision and accuracy for a method.

2.8 Some methods, such as isotope-dilution methods, pH and ignitability, do not use all of the QC elements listed in Section 3.1. Method exceptions to these requirements are listed in the laboratory's analytical SOPs.

2.9 For isotope dilution methods, the assessment of matrix effects on method performance is met with the use of isotopically labelled compounds. These isotopically labelled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance can by judged by recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample.

2.10 Deviations from these QC elements must either be noted in project planning documents (QAPPs, QAPjPs, SAPs, SOWs, QAS, or equivalent) or in a non-conformance memo (NCM).

3. DATA EVALUATION AND CORRECTIVE ACTION

3.1 General Guidelines

3.1.1. Any QC component that is outside of established control limits is considered an out-of-control event. All out-of-control events must be documented and the associated data evaluated. Depending on the specific circumstances, evaluation can lead to a variety of actions. The following sections and the flowcharts describe the appropriate corrective action for the most common QC failures. However, it is not possible to address all possible data evaluation scenarios in this policy. <u>The guiding principle for all evaluations is that the data and corrective action decisions must be defensible using STL Sacramento policies, procedures or scientific evidence, and justified in the project records.</u>

3.1.2. If reanalysis for QC failures is conducted and the second analysis confirms a QC problem that is outside of the laboratory's control, further testing is not necessary. The problem must be documented and the data properly qualified in the project report.

3.1.3. QC failures that are not corrected by reanalysis are documented in NCMs or using an electronic anomaly system as described in SAC-QA-0023.

3.1.4. When ongoing, systematic problems are identified, work must stop until it can be demonstrated that the system is in control again. Utilize the procedures in Attachment II, Section 2, "Examine and Investigate Collected Data (Trend Evaluation)" of this SOP to evaluate data trends.

3.2 Method Blank (MB) Evaluation (also see Figure 1)

3.2.1. Method Blank Acceptance Criteria

The results of the method blank shall be one of the QC measures used to assess batch acceptance. Results are acceptable if all target analyte concentrations in the MB meet the following criteria:

- Analytes reported in the blank are less than 1/10 of the measured of the analyte in the samples in the associated preparation batch, or
- The blank contamination is less than the analyte concentration present in the samples and is less than 1/10 of the regulatory limit, or
- Analytes reported in the method blank are not the anlatyes reported in the associated samples, or
- Analytes levels in the method blank are less than or equal to the reporting limit.

Note: *Positive method blank results slightly below the reporting limit should still be evaluated by the analyst for potential impact on sample results which are at or near the reporting limit.*

3.2.2. Corrective Action for Method Blank Failure

If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. Samples associated with the contaminated blank shall be reprocessed for analysis or, under the following circumstances, may be reported as qualified (qualifier flags or narrative comments):

- MB contamination at a level less than the reporting limit with sample results at levels near the RL, based on analyst's judgement may be flagged, or
- Analyte concentrations in samples are greater than 10 times blank contamination, or
- The contaminant is a common blank contaminant (see Table I) and the MB concentration is less than 5 times the RL.

[1] *The LIMS will automatically add the "B" flag (for organic tests not in the "DIOXIN" department) and "J" flag (for inorganic tests).*

3.2.3.

Projects performed under the auspices of the USACE must meet USACE-specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit for each analyte, or less than 1/10 of the regulatory limit, or less than 1/10 of the sample result for the same analyte, whichever is greater. The concentrations of common laboratory contaminants shall not exceed the reporting limit.

Refer to the DOD QSM Policy (QA-021-SAC) for details of DOD-Specific Method Blank Acceptance criteria and corrective actions.

TABLE I
COMMON LABORATORY CONTAMINANTS

Analyte	Method
Methylene Chloride	Volatile Organics (GC or GC/MS)
Acetone	Volatile Organics (GC or GC/MS)
2-Butanone	Volatile Organics (GC or GC/MS)
Phthalate Esters	Semi-Volatile Organics (GC or GC/MS)
Octachlorodibenzo-p-dioxin (OCDD)	Dioxin Analysis (HRGC/HRMS or HRGC/LRMS)

TABLE I	
COMMON LABORATORY CONTAMINANTS	
Analyte	**Method**
Copper	Metals (ICP or ICP/MS)
Zinc	Metals (ICP or ICP/MS)
Iron	Metals (ICP or ICP/MS)
Lead	Metals (ICP or ICP/MS)

3.3 Laboratory Control Samples (LCS) Evaluation (see Figure 2)

3.3.1. The LCS recovery for the control analytes must be within established control limits. Control analytes may be defined by a project.

3.3.1.1. The analyst should evaluate the non-controlled analyte recovery for possible trends when these analytes fail the control limits. Utilize the procedures in Attachment II, Section 2, "Examine and Investigate Collected Data (Trend Evaluation)" of this SOP to evaluate data trends.

3.3.2. (Effective July 1, 2005) The LCS recovery for all analytes (except for the defined poor performers, Table III) must be within established control limits. Analytes which exceed the control limits are subject to further evaluation. Control limits may be either historically derived, defined by the analytical method, or defined by a project-specific QAPP.

3.3.2.1. In the event that more LCS analytes are spiked than are required to be spiked by a client QAPP, evaluation and control of the LCS is based on the QAPP analyte list only.

3.3.2.2. If analytes are poor performers (as listed in Table III), the recovery is calculated, however, no corrective action beyond documentation is required unless specified in the project documents.

3.3.2.3. If the failed analytes are the clients "analytes of concern" then corrective action is required. The "analytes of concern" may constitute a subset of the requested target analyte list. If poor performing analytes are "analytes of concern" this requirement supersedes section 4.3.2.2.

3.3.2.4. If the number of failed analytes exceeds the number of "marginal exceedences" permitted (Table II), or analytes are outside the marginal exceedence ranges, the LCS is considered failed and corrective action is required.

3.3.2.5. If the allowable number of analytes exceeds control limits based on Table II, they are compared to the marginal exceedence limits. Marginal exceedence limits may be historically derived, defined by the analytical method, or defined by a project-specific QAPP.

3.3.2.6. If the exceedences are within the marginal exceedence limits, and they are determined to be "sporadic", i.e., randomly occurring, rather than indicative of a trend, then the LCS is acceptable and the data may be reported. The exceedence should be noted in an NCM. In general, the same analyte exceeding the LCS control limit two out of the past three LCS is indicative of non-random behavior. Control charts should be reviewed to determine trends.

3.3.2.7. If the exceedences are outside the marginal exceedence limit, or are determined to be a trend rather than randomly occurring, the LCS is not acceptable and corrective action must occur.

TABLE II		
Allowable Marginal Exceedences		
Number of LCS Analytes	**Number of "Marginal Exceedences" permitted**	**Methods in this Range**
11-30	1	PAH-SIM, 8081A, 8290, 8310, 8330,
31-50	2	
51-70	3	8270, 8260
71-90	4	
> 90	5	

Note: Lists with fewer than 11 analytes are not permitted marginal exceedences. This include methods 8015B, 8021B, 8082, and all general chemistry methods. Methods 6010 and 6020 will base the number of permitted marginal exceedences on the number of elements evaluated in the LCS.

3.3.3. Poor Performing Analytes

Poor performing analytes are those analytes identified as not performing well with specific methods. These compounds generally have low percent recoveries and high standard deviations when the results of the LCS are compiled. Specific problems may also be noted by the standard manufacturers. A list of poor performing analytes is in Table III.

TABLE III	
Poor Performing Analytes	
Analysis	**Analyte**
8260	2-Chloroethylvinyl ether
	"Appendix IX" components
8270	Aniline
	4-Chloroaniline[a]
	3-Nitroaniline
	3,3'-Dichlorobenzidine[a]
	Benzoic Acid[a]
	Benzidine
	"Appendix IX" components
8330 (Solid)	Tetryl[a]

[a] *These analytes are noted as poor performing analytes in the DOD QSM, Version 3 Final*

3.3.4. The percent recovery is calculated as follows:

$$\text{LCS Percent Recovery} = \frac{X}{t} \times 100$$

Where: X = observed concentration
t = concentration of spike added

3.3.5. Corrective Action for LCS Failure

- check calculations,
- check instrument performance,
- reanalyze the LCS, and if still outside of control limits,
- evaluate for special circumstances (see below), and/or
- reprepare and reanalyze all samples in the QC batch.

Special Circumstances:

1. In the case where the LCS is high and the analyte of concern is not detected in the samples, it is acceptable to report the data with an anomaly.
2. In the case of volatile analyses or other non-prep methods, if the LCS fails, it may be reprepared and reanalyzed within the same tune period or daily sequence.
3. In the case where all target requested analytes are within control, but some other LCS compounds are out of control, the LCS may still be considered acceptable for reporting.

3.4 Duplicate Laboratory Control Samples (LCS/LCSD) Evaluation (see Figure 2)

3.4.1. The recovery for each spike of the pair must be within established control limits. The formula used to calculate LCSD recoveries is the same as the formula for LCS spike recoveries. If a batch includes samples requiring LCS control and samples requiring DCS control, the reported LCS should be the first LCS analyzed. If either LCS fails, this must be described in the final report case narrative.

3.4.2. The relative percent difference (RPD) for the pair is calculated as follows:

$$\text{RPD} = \left[\frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} \right] \times 100$$

Where: X_1 = first observed concentration
X_2 = second observed concentration

3.4.3. Corrective Action for LCS/LCSD Recovery (Accuracy) Failure

- check calculations,
- check instrument performance,
- reanalyze and/or reprepare and reanalyze all samples in the QC batch.
- The LIMS will automatically flag recovery failures with an "a".

Note: *If either the LCS or the LCSD spike fails and the batch cannot be reanalyzed, the failure must be documented and noted in the final report. Also see notes under Section 4.3.5.*

3.4.4. Corrective Action for LCSD Precision Failure

- check calculations;
- check instrument performance;
- if the RPD is out of control but both accuracy recoveries are within acceptance criteria, prepare an NCM, and qualify report.
- The LIMS will automatically flag precision failures with a "p".

Note: *Because LCS/LCSD limits are based on the standard deviation of data collected over time and include long-term precision, it would be unusual to fail precision limits while meeting accuracy limits. If this occurs with any frequency, either the control limits or the process or both should be reevaluated.*

3.5 Surrogate Evaluation (also see Figure 3)

3.5.1. Surrogate recoveries must be within established control limits. Method QC (MB, LCS, and/or LCSD) results are not acceptable unless the surrogate recoveries for those QC samples are within control limits. If MS/MSD, DU or field samples require dilutions beyond the threshold stated in the analytical SOPs, routine surrogate control limits do not apply and recoveries are not evaluated. This should be noted in the final report.

3.5.2. The LIMS automatically flags out of control surrogate recoveries with an asterisk (*), and adds the footnote that the surrogate is outside the control limits.

3.5.3. The recovery is calculated as follows:

$$\text{Surrogate Percent Recovery} = \frac{X}{t} \times 100$$

Where: X = observed concentration
t = concentration of surrogate added

3.5.4. Corrective Action

3.5.4.1. Surrogate Failures in MB, LCS, or LCSD

- check calculation and instrument performance,
- reanalyze QC sample and/or reanalyze all samples in the QC batch.

Note: *Unless otherwise specified by the client, it may be possible to report qualified results if method QC surrogate recoveries are biased high and analytes were not detected in the field samples. However, all other QC requirements would have to be met and the failure would have to be noted in the final report.*

3.5.4.2. Surrogate Failures in Field Samples or MS/MSD

- check calculation and instrument performance;
- evaluate objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses);
- consult with the client in the event of low bias (as evidenced by low recoveries) in conjunction with possible matrix effect;
- re-analyze or re-prepare the affected samples;
- and document the failure and note it in the final report.

If samples are reprocessed past the holding time, report both original and re-analysis. If results confirm, report both the original and the re-analysis. If the re-analysis/re-preparation is processed within the holding time and the results do not confirm, report the in-control result only.

Note: *Some client programs require reanalysis to confirm matrix interferences. Check special project instructions for this corrective action.*

3.6 Matrix Spike and Matrix Spike Duplicates (MS/MSD) Evaluation (also see Figure 4)

3.6.1. MS and MSD recoveries and RPD should be within established control limits.

3.6.2. If the MS or MSD samples require a dilution beyond the threshold stated in the analytical SOP, routine control limits do not apply and recoveries are not evaluated, but this should be noted in the final report.

3.6.3. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. File an NCM stating that the 4X rule was applied, and report the recovery in the LIMS as "ND MSB". This NCM must be included in the final report.

3.6.4. The MS and MSD recoveries are calculated as follows:

$$\text{MS or MSD Percent Recovery} = \left[\frac{X_s - X}{t}\right] \times 100$$

Where: X = observed concentration in unspiked sample
X_s = observed concentration in spiked sample
t = concentration of spike added

Notes:
1. *If sample result is ND, X = 0 when no values reported below RL.*
2. *If sample result is reported as a value <RL, X = reported value. If <RL values are not reported to the client, then this may appear as "ND" on the final report, but the recoveries will still be corrected.*

3.6.5. The relative percent difference (RPD) for the pair is calculated as described in section 4.4.2.

3.6.6. Corrective Action for MS/MSD recovery or RPD Failure (assuming that the LCS is in control)

- check calculation and instrument performance;
- consider objective evidence of matrix interference (e.g. heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses);
- consider the concentration in the native sample relative to the concentration of spike added; and
- document the failure and note on final report;
- the LIMS will automatically add "a" and "p" flags for failed recovery and precision failures, respectively.

Note: *Some client programs require re-analysis or re-preparation to confirm matrix interferences. Check special project requirements for this corrective action.*

3.7 Sample Duplicate (DU)

3.7.1. The RPD for the sample and its duplicate must be within established control limits. For results which are less than 10 times the reporting limit, the RPD may be outside of established control limits. This should be noted in the final report.

3.7.2. The RPD is calculated as described in section 4.4.2.

3.7.3. Corrective Action for DU Failure

- check calculation and instrument performance,
- document the QC failure and note on the final report.
- check for heterogeneity in the sample.

Note: *If one sample is non-detect the RPD will be 200%. Report as is and note in an NCM.*

4. ESTABLISHING QC ACCEPTANCE LIMITS

4.1 Selecting the Data Set

For new procedures, published method limits can be used until sufficient QC data are acquired (a minimum of 20 data points is required, a minimum of 30 data points is recommended). However, the published limits may not be appropriate if they are based on a single-operator or single-laboratory study. In this case, the QA Manager may establish default limits based on instrument performance and method initial demonstration of capability until enough data is collected for laboratory established limits to be determined. For existing procedures, data collected over several months to a year can be used. Control charts are used together with the calculated mean and standard deviation to determine if the data sets being considered are free of trends and are representative. If it appears that a trend is present, further evaluation of the process is required before limits may be updated. If it appears that the data include gross outliers, outlier tests such as the Grubbs Test, Dixon Test (for 20 or fewer data points), or Rule-of-Huge-Error Test can be used to justify eliminating individual data points. Laboratory established limits must be reevaluated at least annually.

Note: *Refer to Attachment II for a more detailed procedure description.*

4.2 Calculating Laboratory Statistical Performance

Accuracy: mean recovery $\pm 3s$
Precision: zero to (mean RPD + 3s)

Where: s = standard deviation

Control limits for spike analytes are generated using LCS/LCSD data. These limits are used for both LCS and Matrix Spike samples. Control limits for surrogates are generated using both QC and field sample recoveries. If there are insufficient sample surrogate recovery data available to calculate limits, method limits may be used, if available. For methods, matrices, and/or analytes with very limited data, interim limits should be established using available data or by analogy to similar methods or matrices. Collection of points for tests with limited data may span more than a 1 year period.

4.3 Evaluating and Setting Control Limits

4.3.1. The working control limits to be used by the laboratory are based on evaluation of the calculated laboratory statistical performance and available interlaboratory limits provided in the reference methods. Note that some SW-846 methods only supply single-operator or single-laboratory method performance data, which should be used for guidance only and may not be appropriate for routine lab operation.

4.3.2. The control charts should be evaluated for systematic trends and consistency of the performance of the analytical procedure at least annually or whenever new patterns of performance are observed in the laboratory data (i.e. new methods, equipment, etc.), or to evaluate sporadic marginal exceedences (See Attachment II, Section 2).

4.3.3. Current "laboratory-generated limits" should also be compared to the method continuing calibration criteria as outlined in Table IV, below.

Table IV

Criteria for Evaluating Control Limits

Parameter	Minimum SD[1]	Minimum 3SD	Maximum Lower Control Limit	Minimum Upper Control Limit	Minimum RPD
Semivolatile Organics by GC	5	15	Mean – 3SD	Mean + 3SD	15
Volatile Organics by GC	5	15	85	115	15
Semivolatile Organics by GCMS	6.7	20	Mean – 3SD	Mean + 3SD	15
Volatile Organics by GCMS	6.7	20	80	120	15
Metals	3.3	10	90	110	15
Low-Resolution MS (e.g. 8280A)	10	30	Mean – 3SD	Mean + 3SD	20
High-Resolution MS (e.g. 8290)	6.7	20	Mean – 3SD	Mean + 3SD	20
LCMS	6.7	20	Mean – 3SD	Mean + 3SD	15

1 *The minimum 3SD criteria in Table II are based on the method CCV limits (1/3 the method CCV Limit = minimum SD).*

4.3.4. Inorganic non-metals are evaluated in accordance with the reference method(s) for the parameter, as frequently these will have fixed QC limits based on method requirements.

4.3.5. When evaluating current "laboratory-generated limits" against historical "laboratory-generated limits", the laboratory QA manager should investigate any significant

changes in "laboratory-generated mean" and "laboratory-generated range" and should attempt to identify the cause before making any changes to the laboratory limits. If the recalculated limits are consistent with the historical limits, no investigation is required and the limits may be updated in the database.

4.3.6. If a method has specified the control limits to be used, then statistical control limits will be generated and kept on file, but not applied.

Notes:

1. *Laboratory-generated mean = statistical mean (i.e.* $\sum \frac{x_i}{n}$ *).*
2. *Laboratory-generated range = statistical ranges indicated in the previous section*
3. *If the in-house calculated data lead to limits that are significantly tighter than both the guidance limits and/or the calibration acceptance criteria for the method, the laboratory can default to using the laboratory-generated mean ± calibration acceptance limit, for tests with a sample preparation step, or 100% ± calibration acceptance limit for tests without a sample preparation step. Unreasonably tight statistical limits can result from the exclusion of unacceptable results from the database. If investigation demonstrates that this is happening, the laboratory's data entry systems should be improved. The lab should employ a statistical test for outlier values before excluding data points. (Reiterations of outlier tests lead to narrower limits and should be used with caution.)*
4. *If the decision is to use guidance limits from the method, the laboratory should investigate procedural improvements leading to better performance. This is in the case that the control limits which are generated are determined to be too wide or too low for the method capabilities, based on evaluation of past method performance and the method guidance limits.*
5. *If the laboratory-generated mean is within ± 10% of the historical mean, the two means are not significantly different and no investigation of cause of change would be necessary.*

4.4 Control limits are updated on an annual basis. Some regulatory agencies (such as the state of Arizona) require control limits to be updated on a 6 month basis. Control charts should be evaluated more frequently (i.e. quarterly) to monitor for trends.

4.5 Marginal Exceedence Limits

Once control limits are established, marginal exceedence limits are established for tests with greater than 10 analytes. Marginal exceedence limits are calculated as mean recovery ± 4 standard deviations. Compare these limits with the minimums specified in Table V.

Table V

Criteria for Evaluating Marginal Exceedence Limits

Parameter	Minimum SD[1]	Minimum 4SD	Maximum Lower Exceedence Limit	Minimum Upper Exceedence Limit
Semivolatile Organics by GC	5	20	Mean – 4SD	Mean + 4SD
Volatile Organics by GC	5	20	80	120
Semivolatile Organics by GCMS	6.7	27	Mean – 4SD	Mean + 4SD
Volatile Organics by GCMS	6.7	27	73	127
Metals	3.3	13	87	113
Low-Resolution MS	10	40	Mean – 4SD	Mean + 4SD
High-Resolution MS	6.7	27	Mean – 4SD	Mean + 4SD
LCMS	6.7	27	Mean – 4SD	Mean + 4SD

SD and 4SD are based on values from Table IV.

4.6 DOD QSM Evaluation

4.6.1. Once historical control limits and marginal exceedence limits have been established, they must be compared to the DOD QSM, evaluated, and cases where the historical limits exceed the QSM limits tabulated.

4.6.2. Evaluation of the historical limits included comparing the historically derived means and standard deviations to those in the DOD QSM.

4.6.3. The instances where the historical limits exceed the QSM limits are to be reported with each DOD project for evaluation by the DOD project chemist.

5. REPORTING QC DATA

The QC data routinely reported includes the LCS, method blank, surrogate standards and MS/MSD. Matrix QC are reported on a project or client basis, and clients are encouraged to identify on the custody forms specific samples to be used for matrix spiking. If specific samples for spiking are not identified, the laboratory will choose one on a random basis per batch. Client reporting requirements are negotiated and documented as part of the project records. Ultimately, all reporting decisions should accommodate the client's requirements.

Figure 1 - Method Blank Evaluation

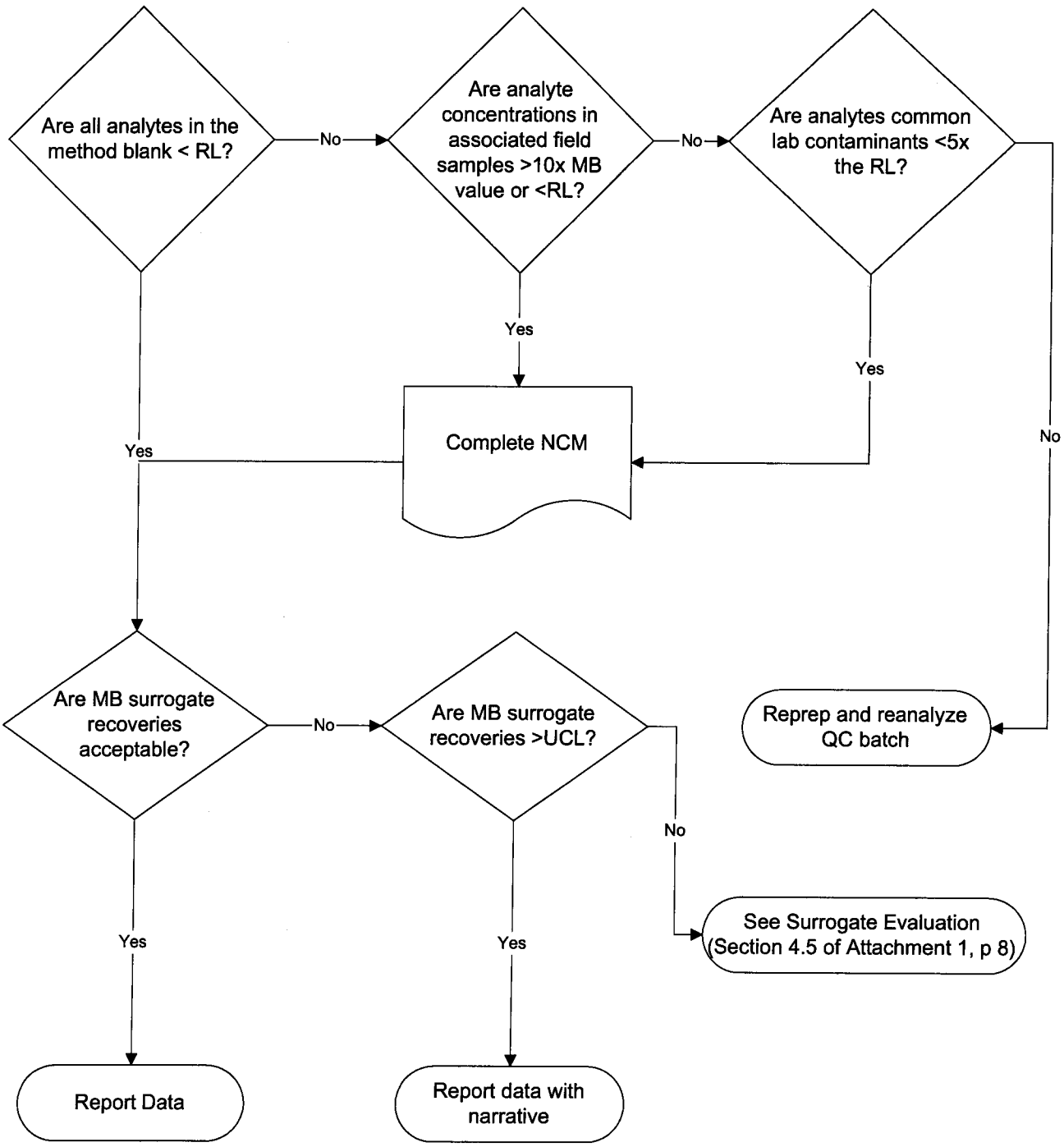


Figure 2 - LCS/LCSD Evaluation

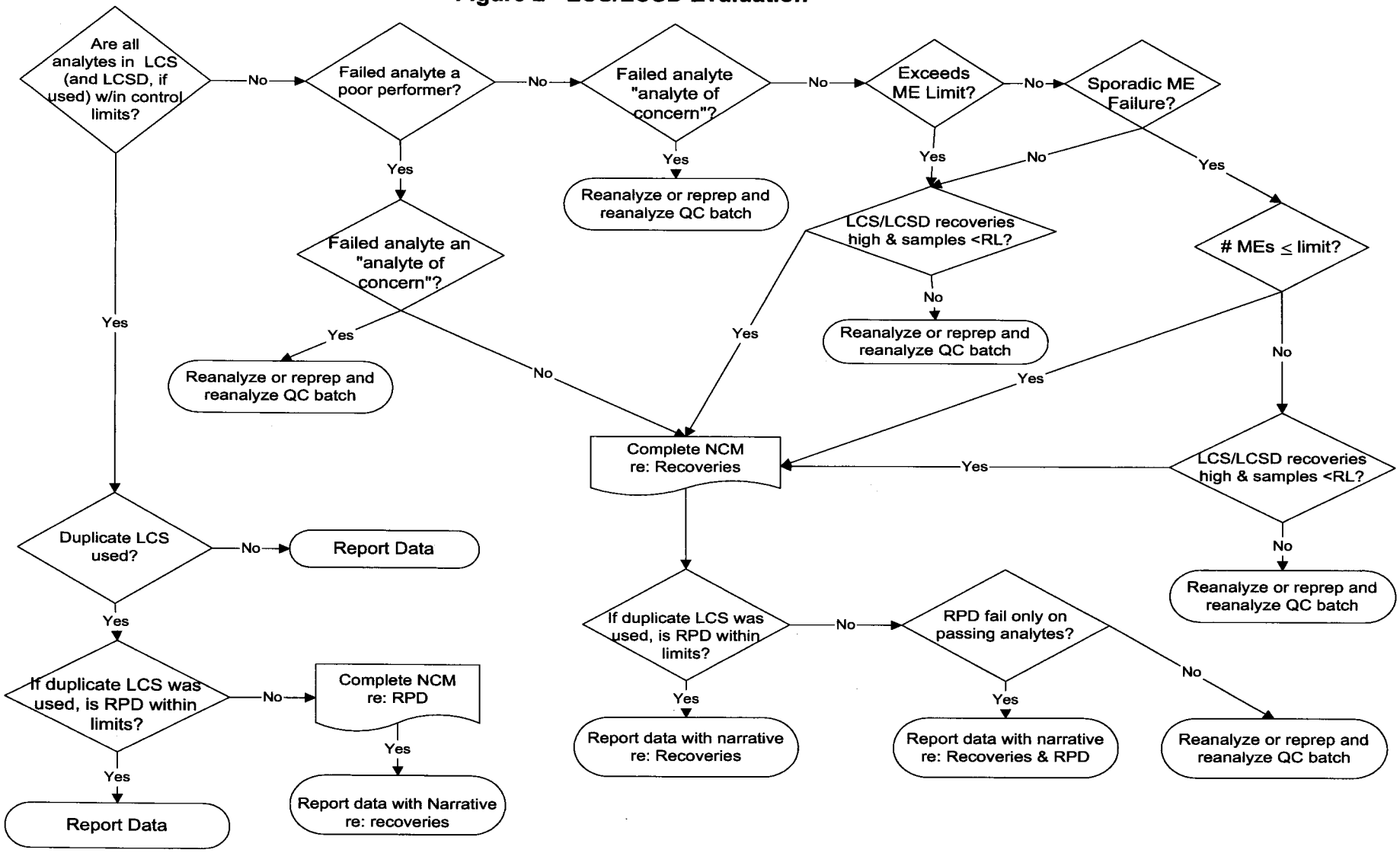


Figure 3 - Surrogate Evaluation

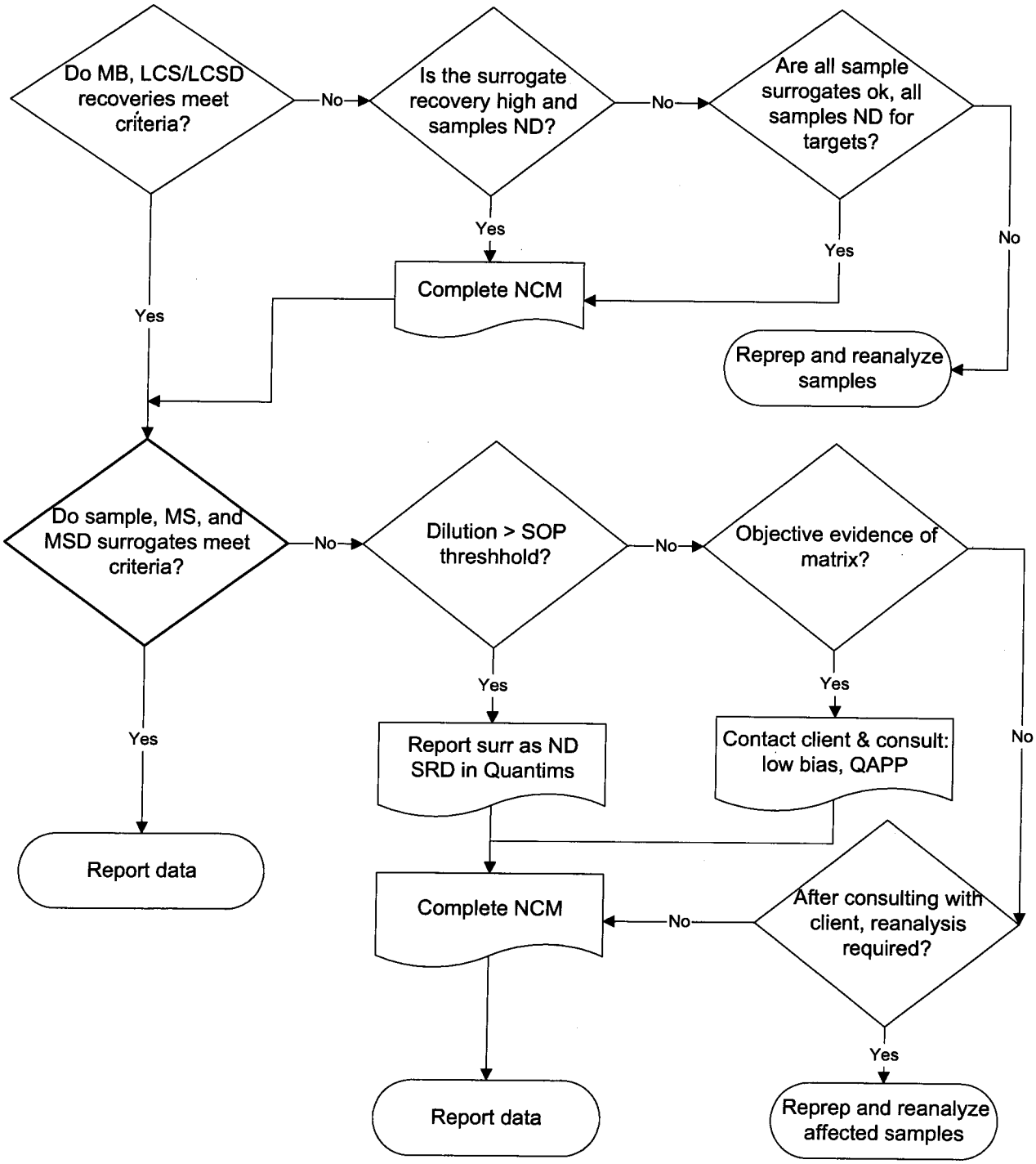
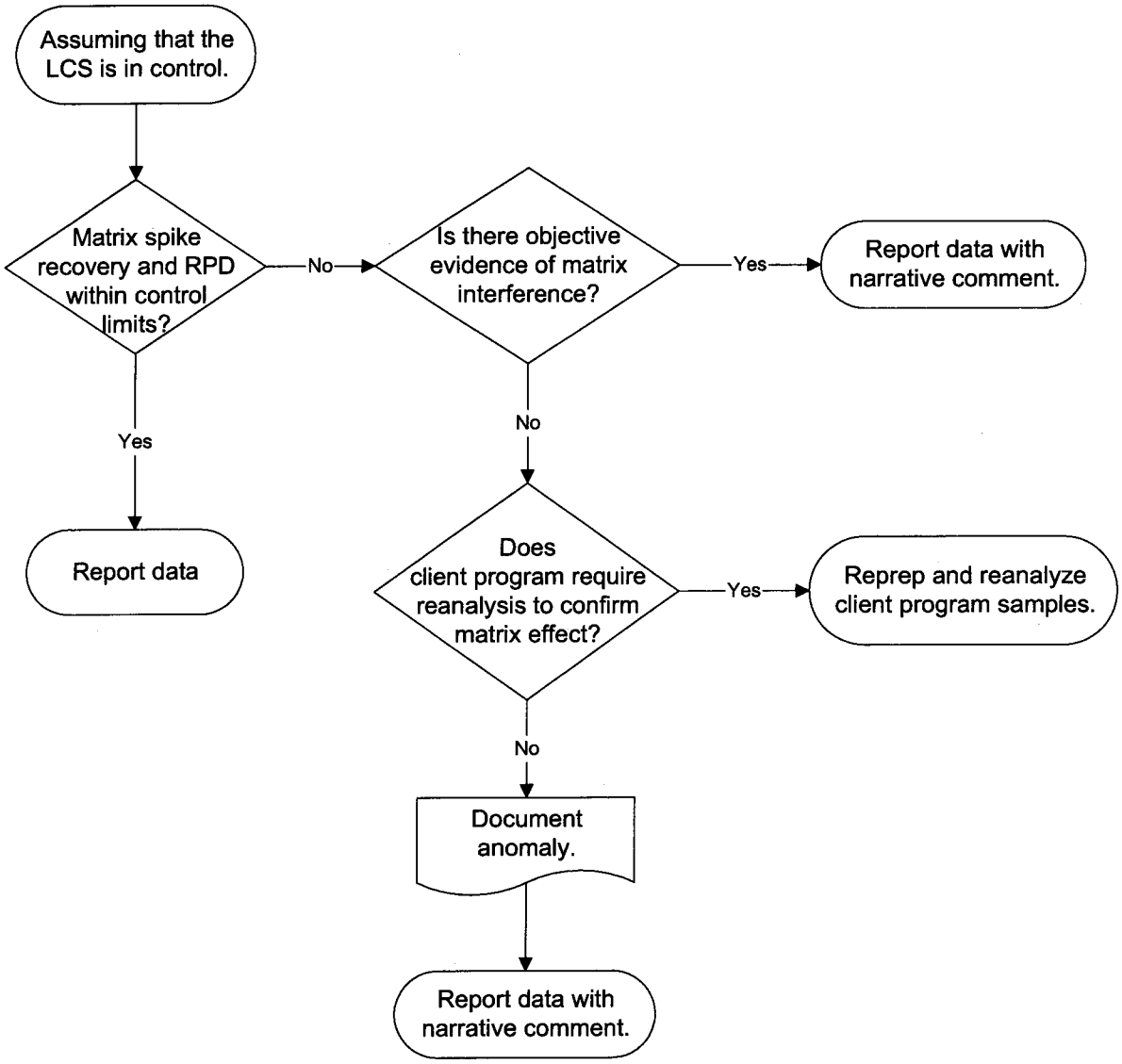


Figure 4 - Matrix Spike/Matrix Spike Duplicate Evaluation



Attachment II
Guidelines for Using TraQar to Generate and Evaluate Control Limits

In accordance with STL Sacramento policy (See the LQM and the main body of this policy), control limits are to be evaluated and updated, as necessary, once a year. Evaluating control charts is an important first step in considering new control limits. This is accomplished using the TraQAr Control Limits program. The program collects a specified set of QC data, performs a Grubbs Outlier Test, calculates 3 standard deviation control limits, compares those to the active ones in QuantIMS, and generates an I-type control chart (ref. ASTM D 6299). The control chart is a plot of results in chronological order to which existing control limits and a mean line have been added. The control chart aids in the examination of the data to be sure that it is representative and appropriate for use in setting new control limits.

1. RUNNING THE CONTROL LIMITS PROGRAM

The TraQAr Control limits program collects data from a database called DataMirror. DataMirror is regularly updated with all QC data that has been loaded into QuantIMS. To maintain performance efficiency, only the most recent 12-18 months of data is present in DataMirror, which effectively limits the period of control charts to data no older than 12-18 months.

1.1 Verify that the location is correctly set.

1.2 Select QA Access Option

This option is only available to QA personnel. It performs control charting, rejection testing and control limit calculating.

1.3 Specify the data to be collected:

1.3.1. QC Type: use the LCS/DCS option to establish both LCS and MS/MSD limits. Use All Surrogates to establish surrogate control limits. The remaining options are rarely used, but may be required to meet client or regulatory needs, or to show trends. For DOD QSM QC limit evaluation, use the "LCS only" surrogate option to generate surrogate limits for clean matrix.

1.3.2. Select a Spike List: Spike lists assocated with the 01, 3W and 3V QC programs in the LIMS have control limits generated and evaluated.

1.4 Select a Representative Time Period: TraQAr will default to 4 months – current date to 4 months previous. While this collects sufficient data most of the time, it may be necessary to widen the range and use one year. Verify that this time range does not overlap the period last used for control limits. The goal is between 35 and 250 points for each parameter within the list.

1.5 Grubbs Outlier Test: Once the data has been collected, TraQAr will perform a Grubbs Test on the data and reject outliers before calculating control limits. It will notify the user when it is complete. (Further information on the Grubbs test may be found in the references).

1.6 Before printing charts and the QC limit report, verify that the outliers discarded should be discarded, and that there are no further outliers within the data. Generally, recoveries greater than 200% for surrogates and 150% for LCS samples are outliers, however, examine this within the context of the data set prior to discarding.

2. EXAMINE AND INVESTIGATE COLLECTED DATA (TREND EVALUATION)

Assuming that an adequate quantity of data points are collected, the next step involves determining that the data set is representative of the lab's performance, and therefore provides a useful prediction of future performance. A key part of the process is examining the data for bias, discontinuities, and trends. Ideally, if conditions are constant over the time period selected and existing limits are appropriate, the data will be evenly distributed around the centerline, with a few points at or slightly outside the control limits. The following are very general guidelines for assessing the representativeness of a data set that does not follow the ideal pattern.

2.1 Bias Relative to Existing Limits ("Mean Shifting")

If a significant majority of QC results falls on one side of the mean line, then investigate the cause. If the cause is a process improvement, then no further action is needed. If the cause is due to a loss of proficiency or reliability, initiate corrective action as appropriate.

2.2 Discontinuous Pattern

If the data appear to run for a period at one mean recovery, and then suddenly jump to a different level, investigate and initiate corrective action as appropriate, and either delay evaluating control limits or excise the out of control period from the current data set.

2.3 Upward or Downward Sloping Pattern

If an upward or downward trend is evident in the data, with no leveling off, there is instability in the method, and reliable control limits cannot be set. In this instance, investigate probable causes and initiate corrective action as appropriate. It may be necessary to delay generating new control limits until the trend has been corrected, and to excise the trending area from the data points used to generate the control limits.

3. ESTABLISHING NEW CONTROL LIMITS

Having collected sufficient data and determined that the data are representative, the next step is to establish new limits. See Section 5.3 of Attachment I of this SOP. Once the limits have been evaluated and any changes made, document any changes and the justification on the control limits report, and initial and date.

4. COMMUNICATING AND IMPLEMENTING NEW CONTROL LIMITS

4.1 Prepare a folder for the control limits. Label with the spike list, the common test ID, and the date the limits were generated.

4.2 Prepare a memo to accompany the folder incorporating the text in the box below. Set the implementation date to be about two weeks in the future.

QC Limit Review
Date:

From: Lisa Stafford, QA Scientist

Spike List ID:

Attached are new QC limits for:

Test Description:

Matrix(es):

Please initial below once you have reviewed the limits, and if more than one person is listed, pass it along to the next in line. When everyone has reviewed the limits, return to QA.

Approvals are due to QA by:

4.3 Send the folder with the memo to the operational manager, then to department manager and other responsible individuals of the area affected by the new limits. The department manager is to review the data and sign the memo to confirm that the data selected are representative of current performance.

4.4 Once the department manager has reviewed the limits and signed the memo, the folder and memo are returned to the QA department.

4.5 On the agreed upon implementation date, update the limits in QuantIMS (Q35), and review the data entry. Record the update date on the folder, and file the data. Send an e-mail to affected analysts, department managers, and operations manager that the limits have been updated, including the new values for the QC limits.

Reviewed 11/08/2007

Title: Document Archiving

Approvals (Signature/Date):

Kym Nelson 3/31/8	Joe Schairer 3/31/08
Kym Nelson — Date	Joe Schairer — Date
Technical Manager	Health & Safety Manager / Coordinator
for: 3/31/2008	3/31/08
Pamela Schemmer — Date	Karla Buechler — Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. SAC-QA-0009.

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1. PURPOSE

1.1. To describe the systems for archiving and retrieval of original documentation.

1.2. The archiving-retrieval system will ensure orderly, traceable archiving and quick retrieval of documents.

1.3. To ensure that all original documentation is retained per the STL Quality Management Plan (QMP) and the STL Sacramento Laboratory Quality Manual (LQM).

2. RESPONSIBILITIES:

2.1. The Archive Custodians are responsible for the organization and upkeep of the archive room, filing and retrieving of appropriately completed containers of data and documents. The Archive Custodians also maintain custody of the key to the Archive Room.

2.2. All employees are responsible for ensuring that all original documents are identified for retention and destruction.

2.3. The record's originator is responsible for the security and integrity of the data or documents up to time of delivery to the Archive Room.

2.4. The originator is responsible for properly packaging, marking and inventorying containers of records to be archived.

2.5. The requester is responsible for the security and integrity of the data or documents after receiving it from the archive room and until returning to the archive room.

3. SAFETY:

3.1. Archive Custodians should use the following safe lifting procedures when handling containers:

- Clear a pathway before moving containers.
- Lift with the legs by bending at the knees and keeping the back straight.
- Test the load. If it is too heavy or awkward, GET HELP.
- Use a cart or hand truck if the container is to be moved more than a few feet.
- Keep the load close to the body.
- Avoid twisting. Perform the lift smoothly and gently.
- Do not attempt to pick up more than one archive box full of data at a time.

3.2 Safety glasses must be on when using the banding machine.

4. PROCEDURE

4.1. Any unauthorized deviations from this procedure must be documented as a non-conformance, with a cause and corrective action described.

4.2. The Archive Custodian or designee monitors the archive room during business hours. The room is locked during non-business hours.

4.3. Initial On-Site Analytical Project Archiving

4.3.1. After the project has been invoiced, it is stored on-site for approximately three (3) months in the archive room.

4.3.2. Inventory the project.

4.3.3. Stack the project with the sample confirmation report on top, followed by:

4.3.3.1. Miscellaneous data.

4.3.3.2. Unused data.

4.3.3.3. Summary report.

4.3.3.4 Raw data.

4.3.4. Rubber band the entire package together. Place a 3" x 3" Post-It® note on the bottom edge of the package with the project number written on the note. The Post-It® is used as a temporary marker/index to facilitate locating projects in the Archive room.

4.3.5. Place the package on the appropriately numbered shelf.

4.3.6. If the project is greater than ten to twelve inches thick, it should stored directly in an archive box. Place the data in the box, put the sample confirmation report on the top of the data, record the project number on the end of the box in the contents area, and write the destruction date (DD) on the end of the box (DD = DDMMMYYYY, such as 20SEP2002). Store the box on the shelf designated for boxed projects from that series of projects.

4.4. Final Analytical Project Preparation

4.4.1. After the project has been stored on-site for approximately three (3) months, or sooner if all of the numbered storage shelves being used to store data are full, the oldest projects are prepared for movement to off-site storage.

4.4.2. Remove **all** rubber bands and paper clips.

4.4.3. Check the inventory of the data package.

4.4.4. Confirm data package arrangement per paragraph 4.3.3.

4.4.5. Place a heavy cardboard divider on the back of the data package. Write project number on the cardboard divider.

4.4.6. Band the data package on the banding machine. Band in two directions, and

ensure the package does not 'curl' while banding.

4.4.7. If the project is greater than ten to twelve inches thick, banding is not necessary. The project will be archived in an archive box by itself. Place the data in the box; put the sample confirmation report on the top of the data. See 4.3.6.

4.4.8. Alternately, small projects may be filed in a 10" x 13" manila envelope. Organize the project as described above, insert the data into the envelope, and close the clasp on the back of the envelope. Write the six digit project number in the upper right hand corner of the front of the envelope, and the destruction date on the front of the envelope. (DD = DDMMMYYYY). As a general rule, however, envelopes should only be used if the analytical project data will fit into a single envelope or the data is so flimsy that it is impractical to use the banding machine.

4.4.9. Once the projects have been packaged as described above, they are stored in archive boxes for movement off-site, or single project boxes are prepared for movement off-site.

4.5. Boxing Analytical Project Packages

4.5.1. If filled with multiple analytical project packages, ensure that all destruction dates are consistent. Write the destruction date on the end of the archive box (DD = DDMMMYYYY). Document destruction dates are defined in the STL QMP.

4.5.2. Record each project number on the end of the archive box in the contents area.

4.5.3. If a single analytical project is in the box, record the project number on the end of the archive box in the contents.

4.5.4. When full, inventory the contents of the box to verify accuracy of labeling. Place container number bar code label on the end of the box and record the container number and inventory on the Records Log form.

4.5.5. Enter inventory and box number online at VanguardVaults.com. Enter box number first then the contents of the box. Submit information and print out confirmation. Staple behind your Records Log form.

4.5.6. Stack the boxes in the "Vanguard Vaults" pickup area.

4.6. Miscellaneous Records Archiving

4.6.1. Archive boxes should contain only one type of record. For example, a box labeled as GC Instrument Calibration Data must be exclusive of MDL Raw Data.

4.6.2. Write the contents on the outside end of the box. Include the Record Series Title, Office of Record and Type of Records. Use enough specific information to easily identify the contents, but do not attempt to list every single item.

4.6.3. Write the range of dates for the information contained in the box.

4.6.4. Determine the destruction date for the information and write it on the end of the box (DD = DDMMMYYYY).

4.6.5. When full, inventory the contents of the box to verify accuracy of labeling. Record the inventory on the Records Log form.

4.6.6. Place a container number bar code label on the end of the box and record the container number on the Records Log form.

4.6.7. Inappropriately labeled boxes will be returned to the originator for correction.

4.6.8. Stack the boxes in the Vanguard Vaults pickup area, and have an Archive Custodian enter the appropriate data online to receive a confirmation and notification. Once notification is received electronically, Vanguard Vaults will schedule pick-up.

4.7. Entering archived material into tracking system.

4.7.1. As material is prepared to move off-site, the archive custodians will enter the appropriate information into the "Vanguard Vaults Database," following procedures outlined in that system.

4.7.2. When archived material is ready to be moved to off-site storage, Archive Custodians will identify the containers within the Vanguard Vaults website for shipment and move the containers to the specified pick-up location. The website gives a confirmation number that is printed out. Upon arrival to the lab, Vanguard Vaults will scan the barcodes on the box. The barcodes are linked to their database and pick-up is verified.

4.8. Requesting materials from Archives.

4.8.1. If the material is on-site, the requester will retrieve data from the archive room. The requester will send e-mail to the archivist identifying which records that are not found on-site. For analytical projects, the only required information is the project number. For Miscellaneous Records, the database is searched for information like the department, range of dates, and any other information which will help identify the specific record.

4.8.2. The Archive Custodian will check the database to determine the location of the requested material. If the material is off-site, a request for retrieval is submitted on-line. Once the requested records are available, the archive custodian will notify the requester using the e-mail system.

4.8.3. The requester will pick the records up from the Archive Room. ALL RECORDS THAT LEAVE THE ARCHIVE AREA MUST BE CHECKED OUT IN THE CHECKOUT LOG. Requestor information is also stored on-line for those items obtained from off-site storage.

4.8.4. When the records are returned to archive, the requestor will check items back in the log, (refer to 4.8.3). Records found on-site will be placed on the correct

location on the shelves. Records for return to off-site are placed in a special location for return.

4.8.5. If returning a record that has been banded, the Archive Custodian will ensure that the cardboard divider is present, and will then re-band the document for transfer back to off-site storage.

NOTE: It is the responsibility of the Requestor to verify that the project is complete before returning the data package for re-archive.

4.9. Adding material to previously archived containers.

4.9.1. Additional data (overlooked material, generated at a later date, follow-up correspondence, etc.) may be added to a project or container which is already archived. If item is now too large for the original container, it is deleted out of the box and added to a new box number.

4.9.2. If the original data or container is still on-site, coordinate with an Archive Custodian to obtain access to the archived material and add as appropriate.

4.9.3. If the original data or container has been moved off-site, complete the Archive Add Request portion of the Report Production Request Form, firmly secure it to the data, and place it in the appropriate basket located in the archive area.

4.9.4. The Archive Custodian will submit any project amendments bimonthly to off-site storage for filing with projects.

4.10. Document Destruction

4.10.1. Each month the Custodian requests a destruction report from Vanguard Vaults. This report indicates all containers and projects that are beyond the required retention date.

4.10.2. The documents in question are placed at a central location at Vanguard. A visual check is performed by the Document Custodian to verify the contents match the software file and that retention dates have expired. Documents are then approved for destruction.

4.10.3. After destruction is complete, Vanguard sends destruction certificate to STL Sacramento indicating container number, contents and actual destruction date.

5. DEFINITIONS

5.1. Lot Number - The unique, 9 digit alphanumeric ID assigned to a given shipment of samples received for laboratory services from a client.

5.2. Sample Confirmation Report - A preprinted form identifying the laboratory, the client, the laboratory project number, and general client sample information.

5.3. Miscellaneous Data - Phone logs, custody documents, submitted reports, invoices, checklists, and raw data generated for a lot number.

5.4. Miscellaneous Records - Documents that are generated by the laboratory but which are not exclusively linked with an assigned project number.

5.5. Unused Data - Raw data that has been reviewed against criteria and has been rejected as unsuitable for reporting to a client.

5.6. Summary Report – Quantims generated data sheets including samples and associated QC.

5.7. Raw Data - Documentation that is generated solely by direct instrument printout, or direct observation.

6. REFERENCES:

6.1. STL Quality Management Plan

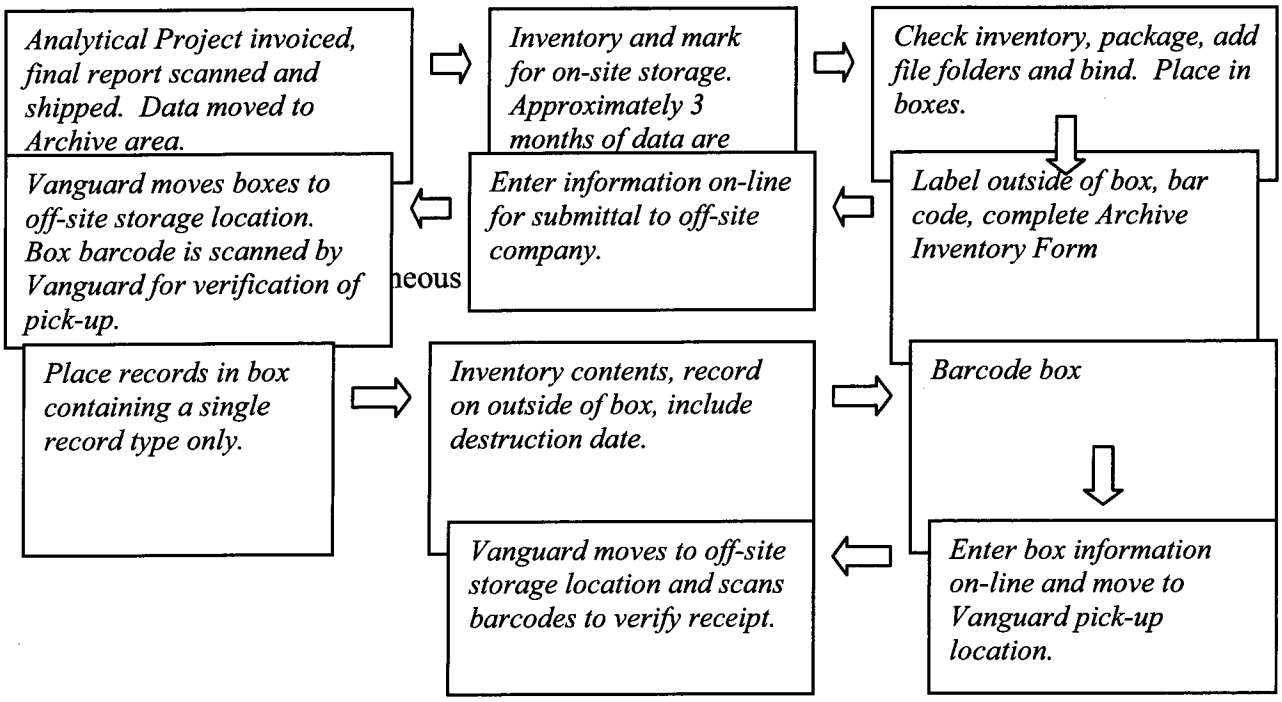
7. MISCELLANEOUS (TABLES, APPENDICES, ETC.):

7.1. Summary of modifications to SOP from previous revisions

7.1.1. This SOP completely revises and supersedes all previous SOPs and documents regarding archiving procedures.

Procedure Flow Diagram

7.2. Analytical Project



Title: SAMPLE RECEIVING, LOGIN, and INTERNAL CHAIN of CUSTODY for AIR SAMPLES

Approvals (Signature/Date):

10/1/07	10/01/2007
Steve Gonzales Sample Control Supervisor Date	William Nash Environmental Health & Safety Coordinator Date
10-2-2007	10/2/07
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1. PURPOSE

1.1. The purpose of this procedure is to ensure the integrity of client samples during sample receipt, login, storage, handling, and analysis.

1.2. This procedure also addresses how project requirements are communicated to the analysts.

2. SCOPE

2.1. This procedure is directed to describe the process by which air samples are received by the laboratory, assessed for acceptability, verified for integrity, logged into the laboratory management system (LIMS), and entered into the laboratory sample stream.

2.2. The procedures herein are set-up to be flexible and versatile due to short holding-time constraints.

3. SAFETY

3.1. 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.

3.2. Specific Safety Concerns and Requirements

3.2.1. Canisters under pressure must be handled with care.

4. DEFINITIONS

4.1. Tedlar bag: A sample container constructed out of an inert, plastic-like material (Tedlar); available in 1-liter, 3-liter, 10-liter, and 25-liter sizes.

4.2. Passivated canister: Commonly referred to as SUMMA canister, SilcoCan, or T.O.-Can in 1.0-Liter, 1.8-Liter, and 6-Liter sizes.

4.2.1. SUMMA Canister: A spherical stainless steel container, which interior has been specially treated by a process (SUMMA passivation) that renders all surfaces inert to volatile organic compounds (VOCs).

4.2.2. SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

4.2.3. T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary electropolishing process and extensively cleaned using an ultrasonic method that ensures a high-quality, passivated surface that maintains the stability of VOCs during storage.

4.3. Vacuum Flow Regulator (VFR): A device which, when connected to a passivated canister, regulates the flow of sample into the canister so that a timed, representative sample can be obtained (also called a composite sample), as opposed to an unregulated, instantaneous sample (grab sample).

4.4. Particulate Filter: A cylindrical stainless steel fitting containing a fritted metal disc, which is connected to the valve of a passivated canister or VFR, to prevent particulate matter from entering and damaging the canister or VFR.

4.5. Pressure Gauge: Device used to measure the vacuum or pressure in a passivated canister. Units of measure range from 30 to 0 inches of mercury (for vacuum) to 0 to 30 psig (for positive pressure). All pressure units are converted to psia.

5. PROCEDURE

5.1. Overview - the sample chain-of-custody (COC) process involves several distinct and important steps:

5.1.1. Samples are received by the laboratory staff and unpacked.

5.1.2. Rented equipment (e.g., passivated canisters, Tedlar bags, VFRs) is checked-in.

5.1.3. Samples are inspected for damage and compared against client COC records.

5.1.4. The project is set-up and samples are logged into LIMS.

5.1.5. Samples are clearly labeled and project and laboratory folders are generated.

5.1.6. Passivated canister samples are vacuum-checked and pressurized.

5.1.7. Samples are transferred to the appropriate storage area. Project documents and paperwork are delivered to the laboratory.

5.2. Sample Receipt

5.2.1. Samples are delivered to the laboratory via several couriers: DHL, UPS, Federal Express, independent courier service, other delivery services or client drop-off.

5.2.2. Upon receipt, the Sample Control personnel will sign the carrier's log (if required) and begin unpacking the samples.

5.2.2.1. Upon receipt at the laboratory, shipping containers (boxes, bags, coolers, ammo boxes, or any other type of container that may contain samples) must be inspected for damage, opened, and the COC reviewed for possible short holding time or quick turnaround time (TAT) samples (< 6 day TAT, Tedlar bags, DTSC passivated canisters).

5.2.2.2. The receiving personnel will record the date, time, and initials on the shipping container. All containers delivered by outside carriers will be noted as being received at the same time.

5.2.2.3. In the event that suspicious odor is present, packages must be unpacked in the fume hood and the laboratory project manager (PM) informed.

5.2.3. A copy of the COC record will be filed in the "Air COC Archive" binder.

5.2.4. Obtain a manila folder and insert a Project Tracking Checklist (PTC) (Figure 6.2, pink sheet). This folder will become the official project folder into which all pertinent paperwork will be kept.

5.2.5. Examine the shipping container for any potential damage. The samples may have also been damaged. Notify PM immediately. Create an electronic nonconformance memo (NCM). Remove all samples, equipment, and documents (e.g., COC forms, air-bill or other shipping documents, Equipment Rental Record, and Canister Field Data Records) from the shipping container. Ensure that paperwork has not slipped between the smaller, internal storage boxes and that small pieces of equipment such as gauges and particulate filters did not get lost within the packing material. Place all documents in the project folder.

5.2.6. Check shipping container for the presence of a Custody Seal. If one is present and intact, circle the "Y" on the PTC in the "Custody Seal Intact" area. If there is no Custody Seal, mark "N/A" on the PTC. If there is a broken seal, inform the PM and document on the PTC.

5.2.7. Examine all samples and equipment for damage. Samples received in Tedlar bags should be checked for leaks by observing if the bag is flat or has very low sample volume. Passivated canister samples should be inspected for broken valves, detached welding, bent valves, dented

bodies, and other evidence of physical damage. An odor emitting from the samples can also indicate possible leakage. Notify PM immediately. Create an electronic NCM.

5.2.8. Verify that all samples recorded on the COC form are present in the shipment. Confirm that the client sample IDs on the COC form match those on the sample labels. Check the COC form for sampler signature and mark "Y" or "N" on the PTC. Any discrepancies and/or missing sampler ID must be reported to the PM and recorded in an electronic NCM.

5.2.9. Important: Until the entire receiving process has been completed, the original COC must remain with the samples in the sample receiving area.

5.2.10. Sign and date the COC form (Figure 6.1) with the date and time the samples were received. In the comments section, record the method of shipment (e.g., Fed-Ex, UPS).

5.2.11. Turnaround times will be calculated from the time and date of sample delivery acceptance. Sample delivery acceptance is the point in time when TestAmerica Los Angeles has determined that it can proceed with the defined work following sample receipt, inspection of the samples, and resolution of any sample discrepancies in COC forms, and project guidance regarding work to be performed.

5.2.12. Initial and date the "Sample Received" section of the PTC (Figure 6.2). Notify project management and analysts if Tedlar bags are received or if **RUSH** analyses have been specified in the accompanying documents.

5.3. Rental Equipment Check-in

5.3.1. If all the equipment being returned is **not** TestAmerica Los Angeles property, skip to Section 5.3.6.

5.3.2. All TestAmerica Los Angeles equipment being returned (passivated canisters, Tedlar bags, coolers, regulators, gauges, etc.) must be checked-in so that proper rental charges can be applied.

5.3.2.1. This step is crucial in the laboratory's ability to inventory its equipment and to seek compensation for any damaged or missing equipment.

5.3.3. The Equipment Rental Record (ERR) is the official document which TestAmerica Los Angeles uses to track and bill all rented equipment (see Figure 6.3). The original ERR is maintained by the laboratory in the "active" file and a copy is sent with the shipment.

5.3.4. When the equipment is returned to the laboratory, the original ERR is retrieved from the active file. To find the correct ERR, retrieve all ERRs pertaining to the client and project. Determine the date the equipment was sent. If the equipment returned are canisters, match the serial number(s) of the passivated canister(s) to the serial number(s) on the ERRs. Each shipment is usually entered in one ERR; larger projects may use more.

5.3.5. When the correct ERR has been obtained, record the return of each piece of equipment received. If all equipment on the ERR has been received, include the ERR with the paperwork that arrived with the samples and **remove the copy from the active file**. Mark returned, unused equipment as "UNUSED", client-designated Do Not Analyze equipment as "DNA," and unusable equipment as "BAD" (i.e., "Low Initial Vacuum"). If there are still outstanding rental equipment, make a copy of the ERR to include with the paperwork and **return the original to the active file**. Notify the PM of any missing or damaged equipment so that the client may be contacted.

5.3.6. Initial and date the "Equipment Checked-in" section of the PTC. If the equipment are non-TestAmerica Tedlar bags or canisters, fill in the quantity received under the "BAGS (Non TA LA)" or "CANS (Non-TA LA)" heading in the comments section. This will alert PMs not to bill the client for equipment.

5.4. Project Set-Up and Log-In

5.4.1. Once the unpacking, inspection, and check-in of all samples have been completed (for ten passivated canisters, the total process is expected to take about 10 minutes), PMs verify project requirements (i.e. analytical methods, report due date, pricing). The Sample Control associate is notified of the quote number to use (from LIMS) to log-in samples. A unique lot number is automatically assigned by LIMS.

5.4.2. Immediately notify the laboratory personnel if there are Tedlar bags or RUSH analyses requested.

5.4.3. Initial and date the "Logged-in" section of the PTC after the samples have been entered into LIMS.

5.5. Labeling of Samples and Generation of Project Folders

5.5.1. Once the samples have been properly set-up and logged-in, all pertinent documents (i.e., report, checklists, COC, ERR, canister certifications, etc.) for the project must be placed in the project folder (manila folder). Copies of these documents will then be distributed to various sections of the laboratory in the appropriate laboratory folders (i.e., orange = GC, blue = GCMS). Sample labels are prepared and

affixed to sample containers. The label must include the following information: work order number, lot number, client sample ID, client name, and passivated canister ID. Also, be sure to label DNA canisters with client name and initial vacuum/pressure.

5.5.2. Initial and date the "Folders to Lab" section of the PTC. Distribute the colored folders to the appropriate groups.

5.6. Canister Pressurization and Leak Check

5.6.1. All passivated canister samples must be checked for initial vacuum/pressure, then pressurized to 10 psig prior to transfer to the sample storage area.

5.6.2. The pressure in the passivated canister must be checked to determine if it has leaked during shipment. The client should record the vacuum or pressure of the passivated canister samples on the Canister Field Data Record (Figure 6.5) prior to shipment to the laboratory. The laboratory staff then checks the vacuum or pressure upon receipt and records the information in the Pressurization Logbook (Figure 6.4).

5.6.2.1. If there is a significant discrepancy between the final field reading and the laboratory reading, the PM is notified. The PM then contacts the client for additional instructions. The information recorded in the Pressurization Logbook is then transferred to the Canister Field Data Record.

5.6.2.2. Samples received with pressure over 5 psig are not pressurized. Samples received with vacuum less than 20 inches of mercury (except those samples identified as "Trip Blanks") are also not pressurized. The PM is notified immediately of the low sample volume. For sub-atmospheric sampling system, if the passivated canister is at atmospheric pressure ("ambient") when the field final pressure check is performed, the sampling period may be suspect. This condition must be checked and documented on the Field Canister Data Sheet upon sample receipt. Further instructions are requested from the client.

5.7. Sample Storage

5.7.1. The final step in the sample receiving process is the transfer of samples to the sample storage area.

5.7.2. Each shelf in the storage area is identified alpha-numerically (i.e., A1, A2, A3...P3, P4). The samples are stored on the shelves based on available space per shelf. The maximum quantity per shelf is 15 of 6-Liter passivated canisters.

5.7.3. Tedlar bags are stored in the bin located in the warehouse AFTER completion of analyses. Tedlar bags are assigned a storage bin by month (i.e., ZZ-1 – January, May, and September; ZZ-2 – February, June, and October; ZZ-3 – March, July, and November; and ZZ-4 – April, August, and December). Tedlar bags are placed in a temporary storage bin in the Sample Control area and the laboratory folder is placed in the "Pending Projects BAGS" file in the laboratory. **Due to the short holding time of Tedlar bags, the Sample Control associate should personally notify the responsible analyst of the receipt of bags** and the analyst should frequently check the backlog or file.

5.8. Canister Storage Blank

5.8.1. Canister storage blanks are used to verify that no cross-contamination has occurred during sample storage.

5.8.2. Every two weeks on Friday, Sample Control associates will prepare one canister storage blank for each canister storage location in the laboratory. A certified clean passivated canister is filled up to 25 psig of Ultra High Purity (UHP) Nitrogen diluent gas. The canister valve and cap are then securely tightened and the passivated canister is transferred to and held in each canister storage location for 14 calendar days.

5.8.3. Canister storage blanks that have been in the storage areas for 14 calendar days will then be replaced and relinquished to the analysts for analysis by EPA TO-15. A COC form will be filled out and these samples will be logged-in.

5.8.3.1. No detected analyte in the canister storage blank may be greater than ½ the reporting limit. A LIMS analytical report and supporting raw data must be submitted to QA within two weeks from being relinquished for analysis.

5.8.3.2. Details of the analysis and corrective actions to be performed when the acceptance criterion for a canister storage blank is not met, may be found in SOP LA-MSA-015.

5.9. Internal Chain of Custody of Samples

5.9.1. Analysts sign-in and sign-out the internal COC as they take and return samples from the sample storage areas.

5.9.2. The check-in of the samples into the storage areas must be recorded in the Internal COC logbook (Figure 6.6), which is generated at log-in by a Microsoft Access database titled “Cradle To Grave”. Analysts who

remove samples from the storage areas must initial and date the "Out" column of the internal COC logbook.

5.9.3. Upon completion of analysis, the samples are returned to the sample storage areas. The return is also recorded in the internal COC. Analysts must initial and date the "In" section of the logbook.

6. RESPONSIBILITIES

6.1. The sample receiving function is primarily the responsibility of the Sample Control associate. Other personnel, such as the PM, data management personnel, analyst, or other trained personnel, serve as a back-up to the Sample Control associate.

6.1.1. The training of all personnel in the proper sample receiving procedures ensures that (1) sample integrity is maintained and (2) samples are received and unpacked quickly so that analytical tests can be performed within the holding time specifications. This is especially critical with the short holding time requirement for the Tedlar bag samples.

6.2. The Sample Control Associate is responsible for ensuring that all personnel are trained in the proper sample receipt and log-in procedures.

6.3. Each analyst is responsible for ensuring that samples are properly received and handled during analysis and that the transfer of samples is traceable.

7. REFERENCES / CROSS-REFERENCES

7.1. TestAmerica Los Angeles SOP LA-MSA-015, current revision, "Determination of Low-Level Volatile Organics in Ambient Whole Air Samples using GC/MS-Scan Mode, Methods EPA TO-14A and EPA TO-15".

7.2. TestAmerica Los Angeles SOP LA-SRA-002, current revision, "Releasing and Cleaning of Sample Canisters, and Cleaning, Calibration, and Setting of Flow Regulators and Vacuum Gauges".

8. ATTACHMENTS

8.1. Attachment 1: Chain of Custody Form

8.2. Attachment 2: Project Tracking Checklist

8.3. Attachment 3: Equipment Rental Record

8.4. Attachment 4: Pressurization Logbook

8.5. Attachment 5: Canister Field Data Record

8.6. Attachment 6: Internal Chain of Custody Form

8.7. Attachment 7: Schematic Representation of Sampling Equipment

9. REVISION HISTORY

9.1. This section has been added beginning with revision 5. Prior revisions are documented in the QA files.

9.2. Changes to revision 4 implemented in revision 5:

9.2.1. Addition (5.3.2.1) made to emphasize that received samples must be checked for short-hold tests immediately upon receipt.

9.2.2. References to NCM system by name (Clouseau) removed.

9.2.3. References to LIMS by name (Quantum's) removed.

9.2.4. References to SILCO canisters removed.

9.2.5. Tedlar bag storage bin nomenclature clarified.

9.2.6. Sections were modified for clerical corrections.

9.3. Changes to revision 5 implemented in revision 6:

9.3.1. This SOP has been formatted using the TestAmerica Corporate QA SOP template.

9.3.2. All references to "Severn Trent Laboratories, Inc." or "STL" have been changed to "TestAmerica".

9.3.3. Changes to section 2 (now section 4 in revision 6), Definitions:

9.3.3.1. The definition of passivated canisters has been added.

9.3.3.2. Reference to the availability of 15-Liter SUMMA canisters has been deleted.

9.3.4. Changes to section 5, Procedure:

9.3.4.1. The statement regarding the transfer of leaking Tedlar bag samples received from clients, into a new and clean Tedlar bag, was deleted from section 5.3.6. The laboratory no longer performs this procedure, unless instructed by the client. Section 5.3.6 has been renumbered as section 5.2.7 in revision 6.

9.3.4.2. The requirement to generate an electronic NCM, when discrepancies are discovered between the COC and the samples received (including sampler's missing ID), has been added to section 5.3.7. Section 5.3.7 has been renumbered as section 5.2.8 in revision 6.

9.3.4.3. The title of section 5.8 has been renamed (from Sample Transfer to Sample Storage). See section 9.3.4.4 below. Section 5.8 has been renumbered as section 5.7 in revision 6.

9.3.4.4. The transfer of samples to the laboratory for analysis (internal chain of custody) was inserted as a separate section on its own. Sections 5.8.3 through 5.8.5 were renumbered under section 5.9 in revision 6.

9.3.4.5. Section 5.9 was deleted. The requirement in this section to state in the report narrative that use of Tedlar bags for Methods EPA TO-14A and TO-15 analyses was a modification to the methods, does not belong to this SOP. This requirement is already stated in the appropriate method SOPs.

9.3.5. Other sections were modified for clerical corrections.

Attachment 1: Chain of Custody Form

Custody Record — **evern Trent Laboratories, Inc.**

STL-4124 (0700)

Client	Project Manager	Date	Chain of Custody Number 024035
Address	Telephone Number (Area Code)/Fax Number	Lab Number	Page ____ of ____
City / State / Zip Code	Site Contact / Lab Contact	Analysis (Attach list if more space is needed)	
Project Name and Location (State)	Carrier/Waybill Number		Special Instructions/ Conditions of Receipt
Contract/Purchase Order/Quote No.	Matrix / Containers & Preservatives		

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Matrix: Air	Aqueous	Sed.	Soil	Containers & Preservatives: Unpres.	H2SO4	HNOS	HCl	NaOH	ZnAc/ NaOH	Analysis	Special Instructions/ Conditions of Receipt

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal
☐ Return To Client ☐ Disposal By Lab ☐ Archive For ____ Months (A fee may be assessed if samples are retained longer than 3 months)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other____

QC Requirements (Specify)

	Date	Time		Date	Time
1. Relinquished By	Date	Time	1. Received By	Date	Time
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments

Attachment 2: Project Tracking Checklist

PROJECT TRACKING CHECKLIST

TestAmerica

QUOTE #: _____

LOT #: _____ CLIENT: _____

SITE/LOCATION: _____

EVENT	INITIAL	DATE	TIME	COMMENTS
Sample Received				ANOMALIES: NONE YES (SEE CLOUSEAU)
*Custody Seals Intact Y N				BAGS (TA LA): 1L 3L 10L
*Sampler Signature Y N				BAGS (NON TA LA): 1L 3L 10L
(* Circle Y or N)				CANS (TA LA): ___ CANS (NON TA LA): ___ REGS: ___
Equipment Checked-In				UNUSED: ___ DNA's: ___
Project Number Assigned				RUSH - ☐24 HOUR ☐48 HOUR ☐72 HOUR
Logged-in				DUE DATE: ___/___/___
Vacuum/Volume Checked				
Folders to Lab				BLUE ORANGE
Typed / Uploaded				
Printed				
Faxed				
ET / JEG Checker				
Coverletter				
Report Approved				
Report Mailed				
Invoiced				

MAILING REMINDERS

PAGINATE ?	NO☐	YES☐	_____
DISK DELIVERABLES?	NO☐	YES☐	_____
CHROMATOGRAMS?	NO☐	YES☐	_____
REPORT COPY TO ANYONE ?	NO☐	YES☐	_____
INVOICE COPY TO ANYONE ?	NO☐	YES☐	_____

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Attachment 3: Equipment Rental Record

Page ____ of ____

TestAmerica

EQUIPMENT RENTAL RECORD

CLIENT:		SHIP TO:	
CLIENT PM:		ATTN.:	
PHONE NO.		PHONE NO.	
ADDRESS:		ADDRESS:	

CLIENT PO# / JOB SITE / PROJECT NAME (REQUIRED TO SHIP):

☐ SOIL GAS SAMPLE ☐ LANDFILL GAS
☐ AMBIENT AIR SAMPLE ☐ INDOOR AIR SAMPLE

ORDERED BY / DATE: _____

VIA:

☐ FED EX ☐ CLIENT PICKUP ☐ COURIER

☐ PRIORITY OVERNITE ☐ STANDARD OVERNITE ☐ ECONOMY (2ND DAY) ☐ X-PRESS SAVER ☐ GROUND

SHIPMENT DATE: _____ RECEIPT DATE: _____

Equipment:

☐ 6-LITER SUMMA CANS: _____ ☐ 1-LITER SUMMA: _____ ☐ REGS: _____ (## MIN/HR) ☐ GAUGE: _____
☐ 1-LITER TEDLAR BAGS: ☐ 3-LITER TEDLAR BAGS: _____ ☐ _____

Comments:

*** UN-RETURNED/DAMAGED CANISTERS / FLOWREGULATORS WILL BE BILLED @ $500.00 EACH ***

CANISTER INFORMATION			VFR INFORMATION		
SERIAL #	DATE RET'D	CONDITION	SERIAL #	DATE RET'D	CONDITION

PRESSURE GAUGES (UN-RETURNED/DAMAGED GAUGES WILL BE BILLED @ $100.00 PER GAUGE)

Serial#	Date Returned	Condition

PARTICULATE FILTERS (UN-RETURNED/DAMAGED FILTERS WILL BE BILLED @ $75.00 PER FILTER)

Quantity out:	Date Returned	Condition

TEDLAR BAGS (UN-RETURNED BAGS WILL BE BILLED @ $15.00 PER BAG)

Quantity/Size	Date Returned	Condition

TUBING ($2.00/FT)		FITTINGS ($5.00 PER FITTING SET)	
TUBING LENGTH:	QUANTITY	TYPE	QUANTITY

Filled by:	Date Sent:

TESTAMERICA Los Angeles will seek full replacement compensation for any equipment lost, stolen, damaged, or un-returned. Contact TESTAMERICA Los Angeles project management immediately if there are any discrepancies with the shipment and the information contained in this RECORD.

1721 South Grand Avenue
Santa Ana, CA 92705
Phone (714) 258-8610 Fax (714) 258-0921

N:\CONERR'S\TESTAMERICA ERRS\TestAmerica ERR GENERIC - 20070620 rev 2.doc

Attachment 4: Pressurization Logbook

TestAmerica Los Angeles CANISTER PRESSURIZATION LOGBOOK

DATE	SAMPLE ID	CAN #	GAUGE CAN READING	INIT. PRESS.	FINAL PRESS.	DIL. GAS	INITIAL	COMMENTS

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Attachment 5: Canister Field Data Record

TestAmerica

CANISTER FIELD DATA RECORD

CLIENT: _____
CANISTER SERIAL #: _____
DATE CLEANED: _____
CLIENT SAMPLE #: _____
SITE LOCATION: _____

VFR ID: _____
Duration of comp. : _____ hrs. / mins.
Flow setting: _____ ml/min
Initials: _____

READING	TIME	Vac (inches Hg) Or PRESS. (psig)	DATE	INITIALS
INITIAL VACUUM CHECK				
INITIAL FIELD VACUUM				
FINAL FIELD READING				
GAUGE READING UPON RECEIPT				

LABORATORY CANISTER PRESSURIZATION			
INITIAL VACUUM (inches Hg and PSIA)			
FINAL PRESSURE (PSIA)			

Pressurization Gas: _____

COMMENTS:

COMPOSITE TIME (HOURS)	FLOW RATE RANGE (ml/min)
15 MIN.	316-333
0.5 Hours	158 – 166.7
1	79.2 – 83.3
2	39.6 – 41.7
4	19.8 – 20.8
6	13.2 – 13.9
8	9.9 – 10.4
10	7.92 – 8.3
12	6.6 – 6.9
24	3.5 – 4.0

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Attachment 6: Internal Chain of Custody Form

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Los Angeles
Internal Chain-of-Custody Log

EXAMPLE

Client: Example Client **Date Received: 01/01/2008** **Project Manager: ABC**

Lab Sample	# Cont.	Container Location	Container Type/ID	OUT int/date/time	IN int/date/time	OUT int/date/time	IN int/date/time	OUT int/date/time	IN int/date/time	RELEASED int/date	CLEAN/DISP int/date

C=Canister TB=Tedlar Bag Vh=VOA w/HCl V=VOA w/o HCl
CGJ=Clear Glass Jar PB=Poly Bottle AGB=Amber Glass Bottle E=Encore

Container Types

Printed on 10/1/2007 11:45

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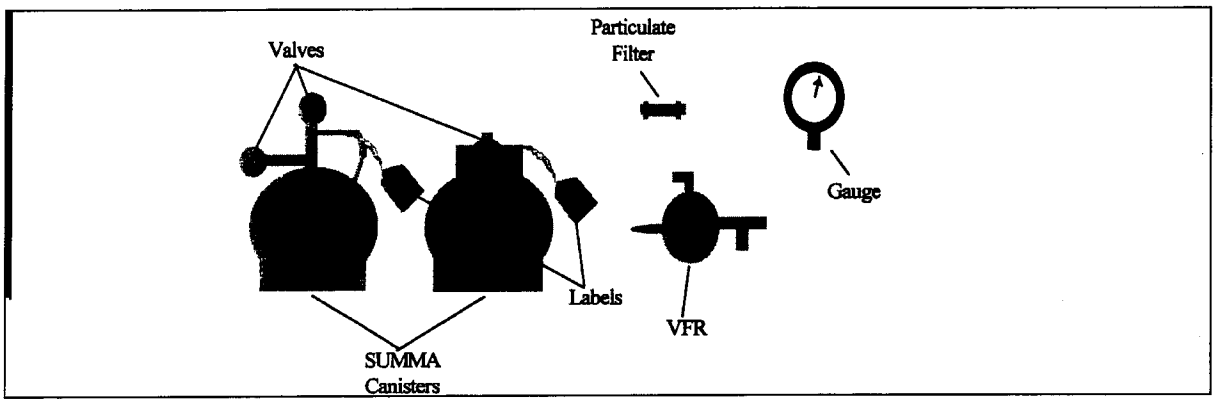
Attachment 7: Schematic Representation of Sampling Equipment



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Los Angeles
FACILITY SOP ATTACHMENT

SOP ID: LA-SRA-001, Rev. 6	**CHANGE FORM ID:** CF1

SOP TITLE: Sample Receiving, Login, and Internal Chain of Custody for Air Samples

REASON FOR ADDITION OR CHANGE: To modify schedule for the preparation of canister storage blank.

CHANGE OR ADDITION: Amend section 5.8 as follows:

5.8 Canister Storage Blank

5.8.1 Canister storage blanks are used to verify that no cross-contamination has occurred during sample storage.

5.8.2 The PM must notify Sample Control in advance when DOD projects are anticipated.

5.8.3 Upon receipt of any DOD project, Sample Control associates will prepare one canister storage blank for each canister storage location in the laboratory. This will be done every two weeks thereafter for the duration of the DOD project. A certified clean passivated canister is first humidified by injecting into the canister 50 uL of nano-pure water. Thereafter, Ultra High Purity (UHP) Nitrogen diluent gas is added until a pressure of 25 psig is attained. The canister valve and cap are then securely tightened and the passivated canister is transferred to and held in each canister storage location for 14 calendar days.

5.8.4 Canister storage blanks that have been in the storage areas for 14 calendar days will then be replaced and relinquished to the analysts for analysis by EPA TO-15. A COC form will be filled out and these samples will be logged-in.

5.8.4.1 No detected analyte in the canister storage blank may be greater than ½ the reporting limit. A LIMS analytical report and supporting raw data must be submitted to QA within two weeks from being relinquished for analysis.

5.8.4.2 Details of the analysis and corrective actions to be performed when the acceptance criterion for a canister storage blank is not met, may be found in SOP LA-MSA-015.

Prepared By: Maria Friedman

***APPROVED BY:**

Technical Specialist

Date 10/9/07

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Los Angeles

FACILITY SOP ATTACHMENT

SOP ID: LA-SRA-001, Rev. 6	CHANGE FORM ID: CF1
Environmental health & Safety Coordinator	Date 10/08/2007
Quality Assurance Manager	Date 10-8-2007
Laboratory Director	Date 10/8/07

*Should be the same signature authorities of SOP being revised.

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Los Angeles

SOP No.: LA-QAS-006, Rev. 4
Effective Date: 04/20/2006

Title: NONCONFORMANCE and CORRECTIVE ACTION SYSTEM

Approvals (Signature/Date):

9-17-2007	9/14/07
Maria Friedman Quality Assurance Manager Date	Elizabeth Winger Laboratory Director Date

This SOP was previously identified as SOP No. SANA-QA-0018.

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STANDARD OPERATING PROCEDURE

TITLE: **NONCONFORMANCE AND CORRECTIVE ACTION SYSTEM**

(SUPERSEDES REV. 3)

Prepared by:	Maria Friedman	
Reviewed by:	Fred Kent, Deputy Technical Director	4/19/06 Date
Approved by:	Maria Friedman, Quality Assurance Manager	4-19-2006 Date
Approved by:	Linda Scharpenberg, Environmental Health & Safety Coordinator	4-19-06 Date
Approved by:	Elizabeth Winger, Laboratory Director	4/19/06 Date

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1. SCOPE AND APPLICATION

1.1. The purpose of this document is to describe procedures for the identification and documentation of nonconformances and the measures taken to correct them. The STL Los Angeles Laboratory Quality Manual (STL LA LQM) requires that a procedure be developed for situations when any aspect of analytical testing and results does not conform to the laboratory's published LQM and standard operating procedures (SOPs) or with agreed requirements of the client.

1.2. All nonconformances (i.e., anomalies and deficiencies) are documented at the time of their occurrence and tracked using a comprehensive database termed "**Clouseau**."

1.3. This document applies to analytical data, services, data reports, and materials purchased by the laboratory or supplied by the laboratory to its clients. Nonconformances related to sample receiving activities must also be documented in the Condition Upon Receipt (CUR) form described in SOP SANA-SC-0001. The nonconformance memo (NCM) system requires immediate notification of the client through the laboratory's project manager (PM) and obtaining instructions on how to proceed.

1.4. Nonconformances can be identified by STL LA employees in the course of their daily operations or by external parties (i.e., customers and representatives of customers) through reviews of records, audits, or proficiency testing.

2. SUMMARY

2.1. This procedure involves the initiation and investigation of root causes of out-of-control occurrences, development of effective corrective actions, and documentation of each step in the process. Nonconformances may relate to client requirements, internal procedural requirements, sample recovery, or equipment issues.

3. DEFINITIONS

3.1. Corrective Action - measure taken to eliminate the causes of an existing defect, nonconformance, or other undesirable situation in order to prevent recurrence.

3.1.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete re-evaluation and restructure of a system.

3.1.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

3.2. Nonconformance - an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation. It is also defined as an unplanned deviation from an established protocol.

3.2.1. An occurrence of a nonconformance, which is the result of STL's actions, is termed a *deficiency*.

3.2.2. An occurrence of a nonconformance, which is the result of events beyond STL's control, is termed an *anomaly*.

3.2.3. Examples of nonconformance

3.2.3.1. Any laboratory quality control (QC) sample (e.g., method blank, laboratory control sample, laboratory control sample duplicate, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrates a systematic deficiency.

3.2.3.2. Any matrix spike or matrix spike duplicate or sample-related QC outside of established control limits attributed to matrix effects must be documented, but an NCM is <u>not</u> required. The matrix-related nonconformances are documented using standard footnotes in the laboratory's data reporting system or the laboratory information management system (LIMS).

3.2.3.3. A practice or procedure is not performed as described according to a client or project document that STL LA has agreed to follow.

3.2.3.4. Purchased materials or services are determined to be defective and their use would affect data quality.

3.2.3.5. ANY holding time violation (HTV) occurs regardless of what or whose actions caused them, including 'ACTS of GOD'.

3.2.4. A formal NCM is not required for routine minor instrument maintenance, malfunctions, and power failures, which can be documented in instrument maintenance logbooks and/or runlogs.

3.3. Other related terms and definitions used in this SOP are provided in the LQM.

4. RESPONSIBILITIES

4.1. Laboratory Associate - All employees are responsible for identifying and documenting nonconformances using an electronic NCM, as soon as they occur. They are also authorized to identify possible measures to correct the problems. Any deviation that might render a measurement suspect shall be documented.

4.2. Supervisor - Each supervisor is responsible for the review and approval of electronic NCMs to ensure that problems adversely affecting data quality are accurately and adequately described and that appropriate personnel are assigned to correct them in a timely manner.

4.2.1. The supervisor must ensure that corrective actions are performed, documented, and supported with evidence of conformance.

4.3. Project Manager - The PM is responsible for communicating project-specific requirements to laboratory staff so that special project requirements are understood and nonconformances are recognized. The PM is the key individual who communicates nonconformance events to clients and documents decisions made with clients. The PM also ensures that corrective actions implemented are appropriate to meet the requirements of a specific project. The PM may withhold final data reports from clients until corrective actions agreed-to with the client have been completed.

4.4. Quality Assurance (QA) Manager - The QA Manager or designee is responsible for reviewing all NCMs to ensure that actions taken are appropriate in ensuring the resolution of QA and QC nonconformances. The QA staff tracks all nonconformances to closure using the Clouseau database. This system is also used to monitor for trends that might indicate long-term quality problems. Systematic problems may be investigated through internal audits to ensure that long-term corrective actions have been successfully completed.

4.4.1. If review of an area reveals a significant problem adversely impacting client data quality, the QA Manager has the authority and responsibility to stop production in that laboratory area.

4.5. Laboratory Director (LD) - The Laboratory Director must emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The LD is also responsible for reviewing NCMs related to customer complaints and for approving the corrective actions that remedy the complaints.

4.6. Corporate QA Director - The STL Corporate Quality Assurance Director should be notified of any systematic nonconformances affecting data quality that are not properly addressed by operations or where the root causes couldn't be identified.

5. SAFETY

5.1. Normal office-dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

5.2. Procedures shall be carried out in a manner that protects the health and safety of all STL LA associates.

5.3. All work must be stopped in case of a known or potential compromise to the health and safety of an STL LA associate. The situation must be reported **immediately** to a supervisor.

6. PROCEDURE

6.1. The NCM procedure involves the following steps:

6.1.1. Evaluation of the significance of the nonconforming work

6.1.2. Investigation to determine the root causes of the problem

6.1.3. Identification of individuals responsible for initiating and/or recommending corrective actions

6.1.4. Specification of how the out-of-control situations and subsequent actions are to be implemented and documented

6.1.5. Specification of the procedures for management to review corrective action reports

6.1.6. Where necessary, the procedure for the notification of the client

6.2. Electronic NCMs

6.2.1. Using Clouseau, STL LA employees are to initiate an electronic NCM whenever procedures, analytical data, services, and data deliverables (hard copy or EDD) deviate from documented laboratory procedures. All nonconformances, with the exception of matrix-related failures,

require an NCM. In addition, all HTVs must be documented using this process. Clouseau may also be used to document *observations*.

6.2.2. Deviation from all laboratory SOPs and QA policies must be documented in an NCM. SOPs and QA policies shall be followed. By signing or initialing laboratory notebooks, bench sheets, data reports, and other quality-related documents, employees are verifying that the SOPs and QA policies have been followed with the exception of pre-approved deviations described in project-specific QA Plans and client checklists.

6.2.2.1. Any intentional deviation from an SOP or a QA policy must be pre-approved by the QA Manager and the appropriate supervisor.

6.2.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple lot numbers and samples, but normally a separate NCM would be initiated for different nonconformance issues. If the nonconformance involves projects for multiple PMs, then the NCM is automatically routed to each PM.

6.3. Creation of NCMs Using Clouseau

6.3.1. While properly logged into a personal computer where Clouseau has been installed, start the Clouseau program.

6.3.2. At the Main Menu, select CREATE. See Attachment A for a screen shot.

6.3.3. Enter the information required at the top of the form. At a minimum, select the Production Area, Classification (anomaly or deficiency), NCM Type, and NCM Description.

6.3.4. For NCMs that affect samples, indicate the relevant batch numbers, lots, sample IDs, and tests by using the ASSOCIATIONS option within Clouseau.

6.3.5. Complete the Comments/Details and Corrective Action sections of the Create NCM Form. These sections may be filled out jointly with the supervisor. Consult the PM or the QA manager if the supervisor and associate are uncertain of corrective actions to be taken. Be objective and specific, but brief. Include enough information that decisions to

approve the NCM can be made easily (include pertinent QC information, e.g., whether or not batch QC data were acceptable).

6.3.5.1. It is imperative that the information entered is accurate and presentable to clients; the information included in the DETAILS section is automatically transferred to the report narrative when the cover letter is created by the PM using a specific program.

6.3.6. Devise corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of possible corrective actions include modifications to nonconforming procedures, repair or replacement of deficient equipment, instrument maintenance, personnel training, instrument calibration, and re-analysis of any affected samples.

6.3.6.1. Where operational corrective actions are required, they shall be supported with reference to QC data, control charts, or other documentation.

6.3.6.2. If the corrective action involves re-training, the training must be documented and submitted to the QA staff before the NCM is considered closed.

6.3.7. Save the NCM using the appropriate command in Clouseau. When presented with the Send E-Mail form, the supervisor of the affected Production Area will be listed in the "Notify Now" box, while affected PMs and QA staff will be listed in the "Notify Later" box. Verify that these names are correct. If any personnel that are not listed should be informed, add their names to the "Notify Now" box by double-clicking on their names in the Personnel box.

6.3.8. Initiate the NCM notification process by selecting SEND E-MAIL.

6.4. Supervisory Review and Approval

6.4.1. The supervisor will receive notification of the NCM via e-mail. The supervisor must log into a computer workstation where Clouseau has been installed, and run Clouseau.

6.4.2. Using the Review NCM form, select the NCM to be reviewed and click on OPEN.

6.4.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas.

6.4.3.1. If the corrective action has not been determined, the situation must be referred to the PM for resolution to ensure client requirements can be satisfied.

6.4.3.2. If the nonconformance is hardware/equipment related, the hardware/equipment shall be tagged as nonconforming and segregated, if possible, to ensure that it is not used until repaired. Refer to section 6.7 for details.

6.4.4. The supervisor will be responsible for the completion and documentation of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is the laboratory's commitment to rectify the problem. The supervisor selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process, which will now route the NCM to the PM.

6.4.4.1. The PM must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for an HTV, any PM must be notified immediately if the assigned PM is unavailable.

6.5. Project Manager Review, Client Notification, and Project Documentation

6.5.1. The PM shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The PM shall indicate using the Client Notification Form in Clouseau the date and method of notification and client's response.

6.5.2. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. Notify the supervisor of any changes made to the corrective action plan.

6.5.3. The PM selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing to QA.

6.5.4. If the nonconformance involves analytical work in process, the <u>final report</u> cannot be released until a PM has approved the NCM.

6.6. Quality Assurance Review and Trending

6.6.1. The QA staff shall review all NCMs for conformance with the LQM and standard laboratory procedures. QA must review the NCMs in a timely manner, generally within 72 hours.

6.6.2. NCMs will be reviewed to ensure that the corrective action was completed, is effective in addressing the root causes of the nonconformance, and will prevent recurrence.

6.6.2.1. If, upon receipt and review of the NCM by the QA staff, it is felt the LD needs to be made aware of the NCM issues, the QA staff will notify the LD using the Under Review/Send E-mail options of Clouseau.

6.6.3. Clouseau's reporting and tracking system will be used to monitor for repetitive failures that might indicate systematic problems. See example chart in Attachment C. Tracking records would (when applicable) include the following information:

6.6.3.1. NCM log number

6.6.3.2. Date initiated

6.6.3.3. Lot number

6.6.3.4. Lab sample ID numbers

6.6.3.5. Method or parameter

6.6.3.6. Nonconformance description

6.6.3.7. Corrective action required

6.6.3.8. Characterization as an anomaly or deficiency

6.6.3.9. Closure of NCM

6.6.4. The QA staff shall review NCMs to identify trends in quality issues that may be systematic in nature and may require long-term corrective actions to prevent recurrence. Recurrent technical problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, and training or replacement of personnel. Findings and

corrective actions from these investigations or audits shall also be documented.

6.6.4.1. Resolution of corrective actions for systematic problems must be documented, along with supporting documents, by the appropriate area supervisor. See section 4.2.1.

6.6.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful. These assessments can be done as part of the internal spot assessments at the laboratory.

6.6.6. The approval of the QA Manager or designee is required in Clouseau to indicate that the NCM has been closed.

6.6.7. QA maintains all electronic NCMs in the Clouseau database.

6.7. Instrument/Equipment Nonconformance Tag

6.7.1. Instruments and equipment that habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a tag indicating the nonconforming condition. See example in Attachment D.

6.7.2. Initiate an electronic NCM and identify the instrument by name and/or identification number.

6.7.3. Corrective action will include permanent removal of the instrument from service or to have the instrument repaired. If an instrument is repaired, its return to control must be demonstrated and documented through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record this information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

7. REFERENCES

7.1. STL LA LQM, current version.

7.2. SOP SANA-SC-0001, *Sample Log In*, current version.

7.3. QA Policy QA-SANA-0011, *Report Revision*, current revision.

7.4. National Environmental Laboratory Accreditation Conference (NELAC), *Quality Systems,* June 2003.

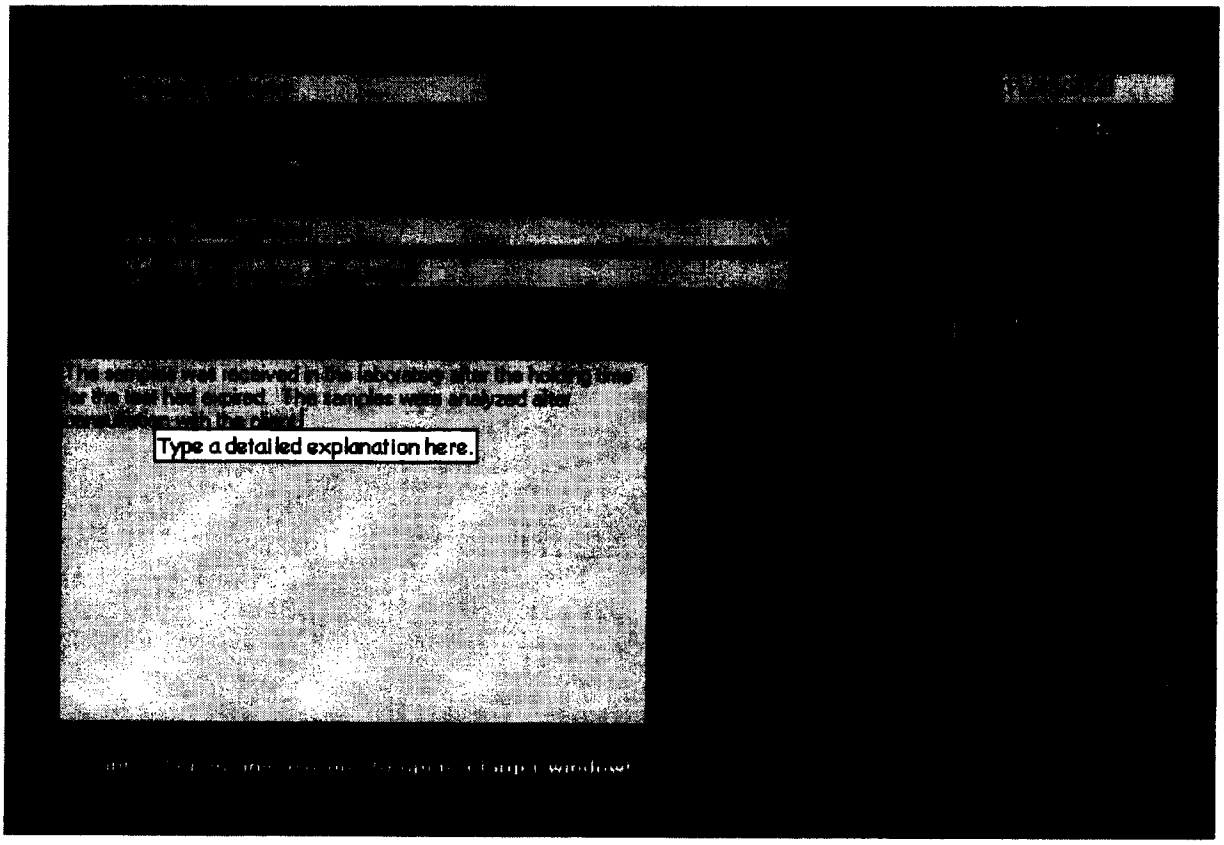
7.5. General requirements for the Competence of Testing and Calibration Laboratories. ISO/IEC 17025.

7.6. Clouseau. Visual Basic Program using Microsoft Access Database, current version.

8. TABLES, DIAGRAMS, AND FLOWCHARTS

ATTACHMENT A

CREATION OF AN ELECTRONIC NONCONFORMANCE MEMO



ATTACHMENT B

EXAMPLE ELECTRONIC NONCONFORMANCE MEMO

Clouseau
Nonconformance Memo

[redacted]

NCM #:	E00836	Classification:	Anomaly
NCM Initiated By:	Gary Beckman	Status:	[redacted]
Date Opened:	08/18/00	Production Area:	GC/MS
Date Closed:	08/18/00	Tests:	8260B
		Lot #'s (Sample #'s):	E0H160253 (6,7)
		QC Batch:	None.

Nonconformance: Sample related issues
Subcategory: Insufficient sample quantity to perform reanalysis

[redacted]

Name	Date	Description
[redacted]		

[redacted]

Name	Date	Corrective Action
[redacted]		

[redacted]

Verified By	Due Date	Status	Notes:
	N/A	Verification not required or requested	This sample condition is outside of lab's control. Document in report narrative.

[redacted]

Name	Date Approved:	Position
Gary Beckman	08/18/00	
Fred Kent	08/18/00	
Paul Christl	08/18/00	
Sevdâ Aleckson	08/18/00	

Date Printed: 08/18/00

Page 1 of 1

ATTACHMENT C

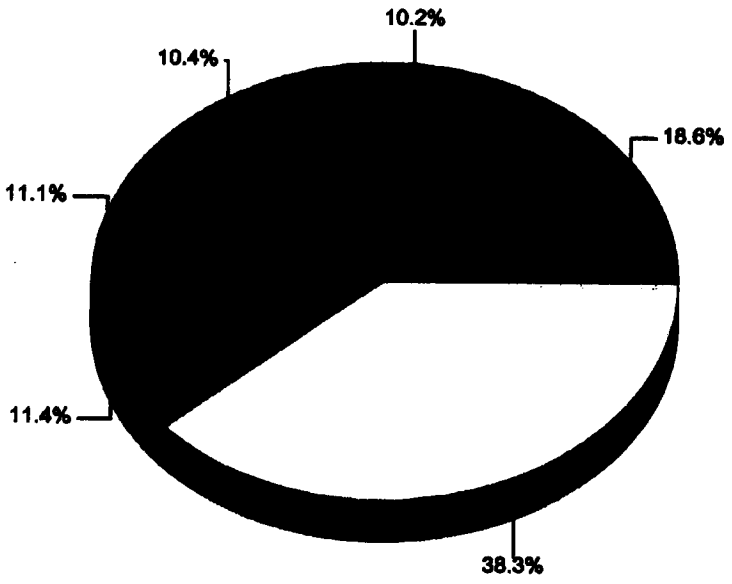
EXAMPLE NONCONFORMANCE TRENDING CHART

Nonconformance Memo (NCM) Chart

For 10/01/00 through 11/03/00

Top 5, By Type, Including Anomalies and Deficiencies

Los Angeles



Insufficient sample volume received from client	38.3%
Sample receiving issues	11.4%
Use of the secondary ion to quantitate	11.1%
Sample description	10.4%
Other (describe in detail)	10.2%
Others	18.6%
Total:	100.0%

ATTACHMENT D

EXAMPLE INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG

STL LOS ANGELES

CAUTION

DO NOT USE

NONCONFORMING ITEM

NCM NUMBER: _____

AFFECTED ITEM: _____

_____

_____

_____ _____
SUPERVISOR DATE

NOTE: WORK MAY NOT PROCEED ON THIS ITEM UNTIL SUCCESSFUL CALIBRATION IS DOCUMENTED.

9. SOP REVISION HISTORY

9.1. This section has been added beginning with revision 4. Prior revisions are documented in the QA files.

9.2. Changes to revision 3 implemented in revision 4:

9.2.1. Section 1.3 was revised to indicate that NCMs related to sample receiving activities must be documented both in the CUR form and in Clouseau.

9.2.2. Section 4.2.1 was added to clarify that supervisors are responsible for ensuring that corrective actions to nonconformances are performed and documented with supporting documentation of conformance.

9.2.3. Reference to the operations/systems manager in section 6.4.3 of the previous SOP revision was deleted. The laboratory does not employ an operations/systems manager.

9.2.4. The last statement in section 6.4.3 (regarding the need to notify LD of the NCM) of the previous SOP revision was moved as section 6.6.2.1 in the current revision.

9.2.5. The last statement in section 6.5.3 (regarding QA's timely review of NCMs) of the previous SOP revision was moved as the last statement in section 6.6.1 of the current revision.

9.2.6. Only the first statement of section 6.5.4 of the previous SOP revision was retained in the current revision. PMs follow a different procedure in notifying QA of revisions to a report. See policy QA-SANA-0011.

9.2.7. Clerical corrections were made to several sections.

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Los Angeles

SOP No.: LA-QAP-002, Rev. 6
Effective Date: 06/25/2007

Title: **TESTAMERICA LOS ANGELES QUALITY CONTROL PROGRAM**

Approvals (Signature/Date):

9-17-2007		9/14/07	
Maria Friedman Quality Assurance Manager	Date	Elizabeth Winger Laboratory Director	Date

This SOP was previously identified as SOP No. QA-SANA-0002.

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

APPROVAL:

6-22-2007
Maria Friedman
Quality Assurance Manager

6/22/07
Elizabeth Winger
Laboratory Director

TESTAMERICA LOS ANGELES
LAB-WIDE

POLICY NAME
TestAmerica Los Angeles Quality Control Program

Supersedes Rev. 5

OBJECTIVE:

This policy describes the program of routine analytical Quality Control (QC) activities. The objective is to generate QC data that demonstrates that the analytical process is in control and that the data meet both client and method requirements. This SOP may be superseded by method- or client-specific requirements.

SCOPE:

This policy is to be enforced and followed throughout the laboratory.

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1. POLICY

1.1. Assessments of QC data relative to control limits determine the acceptability of sample test results. Whenever control criteria are not met, the data must be evaluated to determine appropriate corrective action. The analyst performs the initial evaluation, frequently in conjunction with senior analysts or supervisors. Second-party data reviewers conduct further technical evaluation of the data. Corrective action decisions, particularly whether or not to re-analyze samples, should be done in consultation with the client to the extent possible. Requirements for assessment and corrective action are described in the attachment to this policy.

1.2. TestAmerica's standard QC program is to be communicated to the client prior to acceptance of work. At the same time, the laboratory will work with its clients to understand their special project requirements. Generally, the laboratory's project managers (PMs) serve as liaison between the client and the laboratory to ensure that requirements are properly communicated to both parties. In the event that alternative QC procedures are not specified in a method or by the clients, these standard QC protocols will apply.

1.3. Successful implementation of this QC program requires that it is clearly understood by all TestAmerica Los Angeles associates. Therefore, the essential elements of this program are incorporated in the Laboratory Quality Manual that is reviewed annually by all personnel.

1.4. TestAmerica's QC program as applied to client and regulatory agency requirements:

1.4.1. Resource Conservation Recovery Act (RCRA) and SW-846 Methods - All routine analytical projects performed using SW-846 methods must comply with the requirements described in TestAmerica's Laboratory Quality Manual (LQM) and in Attachment I to this policy. The QC sections of the analytical standard operating procedures (SOPs) referencing SW-846 methods must be consistent with the requirements in Attachment **I.**

1.4.2. Clean Water Act (CWA) and 40CFR Part 136 Listed Methods - Any analytical work conducted in support of a National Pollutant Discharge Elimination System (NPDES) permit or other CWA compliance activities must meet the QC specifications in the TestAmerica LQM. The QC requirements for the specific methods listed in the Quality Assurance Management Plan (QAMP) define the minimum requirements that must be provided in laboratory analytical SOPs.

1.4.3. Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and Contract Laboratory Program (CLP) Methods - Projects performed using USEPA CLP methods must comply with the QC

specifications provided in the TestAmerica LQM. The QC sections of the SOPs referencing these protocols must be consistent with the TestAmerica LQM and the relevant Statement of Work (SOW).

1.4.4. Other Programs or Projects With Clearly Defined QC Requirements

1.4.4.1. The differences between the standard QC program and special project requirements must be specified in project documents. These documents may include Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), Quality Assurance Summaries (QASs), SOPs, contracts, or other approved documents.

1.4.4.2. Documents describing special project requirements must be reviewed and approved by appropriate quality assurance (QA) and operations staff.

1.4.4.3. If the special project requirements appear to result in modifications that contradict federal or state regulatory requirements, the variance must be noted in the project case narrative and communicated to the client. A record of this communication must be retained as a permanent part of the project file.

1.4.4.4. Any special project requirements must be communicated to supervisors, technicians, and analysts in advance of releasing samples for analysis, and the work must be clearly differentiated in the analytical documentation.

1.4.5. Projects Without Specific QC Requirements

1.4.5.1. Any projects, for which no specific QC program is specified, regardless of the source of the analytical methods being used, must follow the requirements in section 2 below.

2. TESTAMERICA LOS ANGELES QUALITY CONTROL PROGRAM

2.1. INTRODUCTION

2.1.1. This QC program is based on the requirements in "Test Methods for Evaluating Solid Waste", USEPA SW-846, Third Edition with promulgated updates. It applies whenever SW-846 analytical methods are used. It also applies in whole or in part whenever project requirements fail to specify

some aspect of QC practices described in this attachment. It does not apply when other well-defined QC programs (e.g., CLP, CLP-like, AFCEE, or NFESC) are specified. This is the base TestAmerica QC program for environmental analyses.

2.1.2. Details concerning instrument calibrations, tunes, and QC that are required for specific methods (e.g., interference check samples for ICP and isotopic spikes for dioxin procedures) are not provided in this attachment. Refer to the appropriate SOPs for information regarding the frequency, assessment, and corrective actions required for additional QC elements.

2.2. DEFINITIONS

2.2.1. QC Batch - The QC batch is a set of up to 20 field samples plus associated laboratory QC samples that are similar in composition (matrix) and are processed within the same time period with the same reagent and standard lots.

2.2.2. Surrogates - Surrogates are organic compounds similar in chemical behavior to the target analytes but are not normally found in environmental samples. Surrogates are added to all samples in a batch to monitor the effects of both the matrix and the analytical process on method accuracy.

2.2.3. Method Blank - The method blank (MB) is a control sample prepared using the same reagents used for the samples. As part of a QC batch, it accompanies the samples through all steps of the analytical procedure. The MB is used to monitor the level of contamination introduced to a batch of samples processed in the laboratory.

2.2.4. Instrument/Calibration Blank - The instrument blank is prepared using the same solvents and reagents (e.g., hexane, methylene chloride, or reagent water) used to dilute the prepared sample extracts or digests. Unlike the MB, it is analyzed without being subject to the preparation steps of the analytical procedure. It is used to monitor laboratory or reagent contamination introduced at the instrumental analysis phase of work. For procedures without a separate preparation or extraction step, an instrument blank is equivalent to the MB, and serves the same purpose.

2.2.5. Laboratory Control Sample - A Laboratory Control Sample (LCS) is prepared using a well-characterized matrix that is spiked, with known amounts of representative analytes. Alternate matrices (e.g., glass beads, washed and baked sodium sulfate) may be used for soil analyses. As part of a QC batch, it accompanies the samples through all steps of the analytical

process. The LCS is used to monitor the accuracy of the analytical process independent of possible interference effects due to sample matrix.

2.2.6. Duplicate Control Sample - A duplicate control sample (DCS) pair, also called the LCS/LCSD, consists of a pair of LCSs analyzed within the same QC batch to monitor precision and accuracy, independent of sample matrix effects.

2.2.7. Matrix Spike and Matrix Spike Duplicate

2.2.7.1. Matrix Spike - A matrix spike (MS) is a replicate portion of one sample in the QC batch that is spiked with known amounts of target analytes. An MS is spiked with the same analytes that are added to the LCS. As part of the QC batch, it accompanies the samples through all steps of the analytical process.

2.2.7.2. Matrix Spike Duplicate - A matrix spike duplicate (MSD) consists of an additional portion of the same sample used to prepare the MS. This portion is spiked and processed exactly like the MS.

2.2.7.3. The MS and MSD results are used to determine the effect of the sample matrix on the precision and accuracy of results. Due to the potential variability of the matrix of each sample, the MS and MSD results may not have immediate bearing on any samples except the one spiked.

2.2.8. Sample Duplicate - A sample duplicate is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision measurements for other samples in the batch.

2.3. BATCH QC ELEMENTS & BATCH PROCESSING

2.3.1. A QC batch is designed to establish freedom from contamination, accuracy, and precision of results for each group of samples, twenty or fewer. With some exceptions as described in sections 3.6 through 3.8 below, the minimum QC elements for each QC batch are as follows:

2.3.1.1. One MB

2.3.1.2. One LCS

2.3.1.3. One MS

2.3.1.4. One MSD

2.3.2. The identity of each QC batch must be documented and traceable, i.e., each batch of field samples must be clearly associated with the applicable QC samples.

2.3.3. To the extent possible, samples that require a preparation step should be analyzed together with their associated QC samples. If the samples in a given QC batch require separate analytical runs, the batch QC in each run, at a minimum, must consist of an acceptable MB or calibration blank. QC samples should not be analyzed independent of the field samples on a different instrument.

2.3.3.1. In the event that extracted samples must be analyzed on different instruments, the MB, at a minimum, must also be analyzed with the samples.

2.3.3.2. In the event that multiple batches are analyzed on different instruments, one valid MB from a single batch may be analyzed to prove that the instrument is not contributing contamination to the samples. This may only be performed with the supervisor's approval. All MBs from all batches must be analyzed. A single MB analysis may not be used to report analytical results from multiple batches.

2.3.4. For analytical procedures that do not include separate extraction or digestion (e.g., volatile organic analysis by purge and trap), the QC batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event, i.e., the same calibration curve, calibration factors or response factors (RFs) must be in effect throughout the analysis.

2.3.5. Field QC samples (e.g., trip blanks, equipment rinsates, and field duplicates) count as individual samples, i.e., they add to the QC batch count. Samples, which require simple re-analysis (e.g., dilutions to adjust a sample extract to the working range of the instrument), do not count as additional samples in the QC batch as long as they are analyzed within the same calibration event.

2.3.6. MS/MSD pairs are not the only acceptable means of demonstrating precision.

2.3.6.1. As requested by clients or required by some methods, batch precision may also be demonstrated through the analysis of sample duplicates. However, the client should be advised that a sample duplicate is less likely to provide usable precision statistics, depending on the likelihood of finding concentrations below reporting limits (RLs).

2.3.6.2. An LCS/LCSD (also DCS) pair may be used to demonstrate batch precision when the client has not supplied sufficient sample to prepare an MS, MSD, or sample duplicate.

2.3.6.3. On-going monitoring of LCS results can be used to determine long-term, between batches, precision control.

2.3.7. Some methods (pH and ignitability, for example) do not use all of the QC elements listed in section 3.1. Method exceptions to these requirements are listed in the QC tables in TestAmerica's LQM and in the laboratory's analytical SOPs.

2.3.8. Deviations from these QC elements must be noted either in the project planning documents (QAPPs, SAPs, SOWs, QAS, or equivalent) or in a nonconformance memo (NCM).

2.4. DATA EVALUATION AND CORRECTIVE ACTION

2.4.1. General Guidelines

2.4.1.1. Any QC component that is outside of established control limits is considered an out-of-control event. All out-of-control events must be documented and the associated data evaluated. Depending on the specific circumstances, evaluation can lead to a variety of actions. The following sections and the flowcharts describe the appropriate corrective actions for the most common QC failures. However, it is not possible to address all possible data evaluation scenarios in this policy.

NOTE: As a guiding principle for all evaluations, the data and corrective action decisions must be defensible using TestAmerica

policies, procedures or scientific evidence, and must be justified in the project records.

2.4.1.2. If re-analysis due to QC failures is conducted and the second analysis confirms a QC problem that is outside of the laboratory's control, further testing is not necessary. The problem must be documented and the data properly qualified in the final report.

2.4.1.3. QC failures that are not corrected by re-analysis are documented in NCMs as described in SOP SANA-QA-0018. Because QC failures due to sample matrix interferences (particularly MS, MSD and surrogate failures) are a function of the method used and are normally outside of the laboratory's control, trending via NCMs is not required. Other forms may be used to document matrix QC failures.

2.4.1.4. When ongoing, systematic problems are identified, work must stop until it can be demonstrated that the system is in control again.

2.4.2. Method Blank Evaluation (see Figure 1)

2.4.2.1. Acceptance Criteria - All analyte concentration in the MB must be ≤ project RL.

2.4.2.2. Corrective Actions for MB Failure - The initial corrective action requirement is the re-analysis of the MB. If not resolved, follow with re-preparation and re-analysis of all samples and QC in the QC batch. The following special situations, while requiring documentation, may allow qualified data to be reported without re-analysis:

2.4.2.2.1. Analyte concentration found in samples is <RL.

2.4.2.2.2. Analyte concentration found in samples is >10x MB concentration.

2.4.2.2.3. The analyte is a common laboratory contaminant (see below) and the MB concentration is <5x RL for organic analyses, or <2x RL for inorganic analyses.

Common Laboratory Contaminants:

Analyte	Method
Methylene Chloride	Volatile Organics (GC or GC/MS)
Acetone	Volatile Organics (GC or GC/MS)
2-Butanone	Volatile Organics (GC or GC/MS)
Phthalate Esters	Semivolatile Organics (GC or GC/MS)
Copper	Metals (ICP or GFAA)
Zinc	Metals (ICP or GFAA)
Iron	Metals (ICP or GFAA)
Lead	Metals (Trace ICP or GFAA)

2.4.3. Laboratory Control Samples (LCS) Evaluation (see Figure 2)

2.4.3.1. Acceptance Criteria -- The LCS recovery for the representative analytes must be within established control limits. The percent recovery is calculated as follows:

$$\text{LCS Percent Recovery} = \frac{X}{t} \times 100$$

Where: X = observed concentration
t = concentration of spike added

2.4.3.1.1. Corrective Actions for LCS Failure

2.4.3.1.2. Check calculations.

2.4.3.1.3. Check instrument performance.

2.4.3.1.4. Re-analyze the LCS.

2.4.3.1.5. Re-prepare and re-analyze all samples in the QC batch.

Note: It is possible to report the data with qualifiers if the LCS recovery is high and analytes were not detected in the samples. Successful MS/MSD and surrogate recoveries may also provide

evidence of control of accuracy, but this would be decided on a case-by-case basis and only with client approval.

2.4.4. Duplicate Laboratory Control Samples (LCS/LCSD or DCS) Evaluation (see Figure 2)

2.4.4.1. Acceptance Criteria - The recovery for each spike of the pair must be within established control limits. The formula used to calculate LCSD recoveries is the same as the formula for LCS spike recoveries. To maintain consistency for mixed batches with projects requiring a single LCS and projects requiring duplicate LCSs, the first spike of the pair is the only one that can be reported as the LCS. The relative percent difference (RPD) for the pair is calculated as follows:

$$\text{RPD} = \left[\frac{X_1 - X_2}{\frac{(X_1 + X_2)}{2}} \right] \times 100$$

Where: X_1 = first observed concentration
X_2 = second observed concentration

2.4.4.2. Corrective Actions for LCS/LCSD Recovery Failure

2.4.4.2.1. Check calculations.

2.4.4.2.2. Check instrument performance.

2.4.4.2.3. Re-analyze and/or re-prepare and re-analyze all samples in the QC batch.

2.4.4.3. If either the LCS or the LCSD spike fails and the batch cannot be re-analyzed, the failure must be documented and noted in the final report. This also applies to a project in a mixed batch (single-LCS-only project samples batched with LCS/LCSD project samples) that only requires reporting a single LCS.

2.4.4.4. Corrective Actions for LCSD Precision Failure

2.4.4.4.1. Check calculations.

2.4.4.4.2. Check instrument performance.

2.4.4.4.3. If MS/MSD RPD is in control, prepare an NCM & qualify report.

2.4.4.4.4. Re-prepare and re-analyze all samples in the QC batch.

2.4.4.5. Since LCS/LCSD limits are based on the standard deviation of data collected over time and does include long-term precision, it would be unusual to fail precision limits while meeting accuracy limits. If this occurs with any frequency, control limits should be re-evaluated.

2.4.5. Surrogate Evaluation (see Figure 3)

2.4.5.1. Acceptance Criteria - Surrogate recoveries must be within established control limits. Method QC (MB, LCS, and/or LCSD) results are not acceptable unless the surrogate recoveries for those QC samples are within control limits. If MS/MSD, sample duplicate, or field samples require dilutions beyond the threshold stated in the analytical SOPs, routine surrogate control limits do not apply and recoveries are not evaluated. This should be noted in the final report. Surrogate recovery is calculated as follows:

$$\text{Surrogate Percent Recovery} = \frac{X}{t} \times 100$$

Where: $X =$ observed concentration
$t =$ concentration of surrogate added

2.4.5.2. Corrective Actions for Surrogate Failure in MB, LCS, or LCSD

2.4.5.2.1. Check calculations and instrument performance.

2.4.5.2.2. Re-analyze QC samples and/or re-analyze all samples in the QC batch.

2.4.5.3. Unless otherwise specified by the client, it may be possible to report qualified results if method QC surrogate recoveries are biased high and analytes were not detected in the field samples. However, all other QC

requirements must be met and the surrogate failure noted in the report.

2.4.5.4. Corrective Actions for Surrogate Failure in Field Samples or MS/MSD

2.4.5.4.1. Check calculations and instrument performance.

2.4.5.4.2. Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses). Otherwise, re-analyze sample to confirm matrix interference, especially if required by a project-specific program.

2.4.5.4.3. Document the failure and note in the final report.

2.4.6. Matrix Spike and Matrix Spike Duplicate (MS/MSD) Evaluation (see Figure 4)

2.4.6.1. Acceptance Criteria - MS and MSD recoveries and RPD should be within established control limits. If MS or MSD samples require dilutions beyond the threshold stated in the analytical SOPs, routine control limits do not apply and recoveries are not evaluated, but this should be noted in the final report. The MS and MSD recoveries are calculated as follows:

$$\text{MS or MSD Percent Recovery} = \left[\frac{X_s - X}{t}\right] \times 100$$

Where: X = observed concentration in unspiked sample
X_s = observed concentration in spiked sample
t = concentration of spike added

Notes:
If sample result is ND, $X = 0$ when no values reported below RL.
If sample result is reported as a value <RL, X = reported value.
CLP forms software uses observed recovery, not concentrations.

$$\text{RPD} = \left[\frac{X_1 - X_2}{\frac{(X_1 + X_2)}{2}} \right] \times 100$$

Where: X_1 = first observed concentration
X_2 = second observed concentration

2.4.6.2. Corrective Actions for MS/MSD Failure (assuming that the LCS is in control)

2.4.6.2.1. Check calculations and instrument performance.

2.4.6.2.2. Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses). Otherwise, re-analyze sample to confirm matrix interference, especially if required by a project-specific program.

2.4.6.2.3. Document the failure and note in the final report.

2.4.7. Sample Duplicate

2.4.7.1. Acceptance Criteria - The RPD for the sample and its duplicate must be within established control limits. The RPD is the same as for the MS/MSD (see section 2.4.6.1).

2.4.7.1.1. Corrective Actions for Sample Duplicate Failure

2.4.7.1.2. Check calculations and instrument performance.

2.4.7.1.3. Document the QC failure and note in the final report.

2.5. ESTABLISHING QC ACCEPTANCE LIMITS

2.5.1. Selecting the Data Set

2.5.1.1. For new procedures, published method limits can be used until sufficient QC data are acquired (minimum of 20 to 30 data points recommended). However, the published limits may not be appropriate if they are based

on a single-operator or single-laboratory study. For existing procedures, data collected over several months to a year or more can be used. Control tables or control charts are used together with the calculated mean and standard deviation to determine if the data sets being considered are free of trends and contain representative measurements. If it appears that the data include gross outliers, outlier tests such as the Q-Test or Rule-of-Huge-Error Test can be used to justify eliminating individual data points. Laboratory-established limits must be re-evaluated annually.

2.5.2. Calculating Laboratory Statistical Performance

2.5.2.1. LCS or LCSD Accuracy - All methods: mean recovery $\pm 3s$

2.5.2.2. Surrogates Accuracy - All methods: mean recovery $\pm 3s$

2.5.2.3. MS/MSD Accuracy

2.5.2.3.1. 8000 series methods: mean recovery $\pm 2s$

2.5.2.3.2. Inorganic parameters: mean recovery $\pm 3s$

2.5.2.4. MS/MSD and Sample Duplicate Precision

2.5.2.4.1. 8000 series methods: Zero to (mean RPD + 2s)

2.5.2.4.2. Other Methods: Zero to (mean RPD + 3s)

2.5.2.5. LCS/LCSD Precision - All methods: Zero to (mean RPD + 3s)

Where: s = standard deviation

Note: If there is insufficient MS/MSD data available to calculate limits, LCS/LCSD limits may be used.

2.5.3. Setting Control Limits

2.5.3.1. The working control limits to be used by the laboratory are based on the evaluation of the calculated laboratory statistical performance and available inter-laboratory limits provided in the reference methods. Note that

some SW-846 methods only supply single-operator or single-laboratory method performance data, which may not be appropriate.

2.5.3.2. Accuracy Evaluation

Mean Evaluation	Range Evaluation	Accuracy Decision
laboratory-generated mean > guidance mean	laboratory-generated range > guidance range	Use laboratory-generated mean & guidance range
laboratory-generated mean > guidance mean	laboratory-generated range < guidance range	Use laboratory-generated mean & laboratory-generated range
laboratory-generated mean < guidance mean	laboratory-generated range > guidance range	Use guidance mean & guidance range
laboratory-generated mean < guidance mean	laboratory-generated range < guidance range	Use guidance mean & laboratory-generated range

2.5.3.3. Precision Evaluation

Range Evaluation	Precision Decision
laboratory-generated range > guidance range	Use guidance range
laboratory-generated range < guidance range	Use laboratory-generated range

Notes: Laboratory-generated mean = statistical mean (i.e. $\Sigma x_i / n$)
Laboratory-generated range = statistical ranges indicated in the previous section
Published guidance range = (upper control limit - lower control limit)
Published guidance mean = (guidance range / 2) + lower control limit

2.5.3.4. If the laboratory-generated mean exceeds 110%, then the guidance mean should be used for the acceptance criteria.

2.5.3.5. If the laboratory-generated mean is within ±5% of the guidance mean, the two means are not significantly different, and thus the laboratory-generated mean is used.

2.5.3.6. If the decision leads to limits that are significantly tighter than both the guidance limits and the calibration acceptance criteria for the method, the laboratory can default to using the laboratory-generated mean ±

calibration acceptance limit. Unreasonably tight statistical limits can result from exclusion of outliers from the database. If investigation demonstrates that this is happening, the laboratory's data entry systems should be improved.

2.5.3.7. If the decision is to use guidance limits from the method, the laboratory should investigate procedural improvements leading to better performance.

3. REPORTING QC DATA

3.1. The QC data routinely reported include the LCS/LCSD, MB, and surrogate recovery. Matrix QC is reported on a project or client basis. Clients are encouraged to identify on the chain of custody forms the specific samples to be used for matrix spiking. Client reporting requirements are negotiated and documented as part of the project records. Ultimately, all reporting decisions should accommodate the client's requirements.

4. REVISION HISTORY

4.1. This section has been added beginning with revision 5. Prior revisions are documented in the QA files.

4.1.1. Changes to revision 4 implemented in revision 5:

4.1.1.1. The last row in the last column in table in section 5.3.2 was modified to correct the accuracy decision from the use of guidance mean and guidance range to the use of guidance mean and laboratory-generated range.

4.1.1.2. All other sections were modified only for clerical corrections.

4.1.2. Changes to revision 5 implemented in revision 6:

4.1.2.1. The referenced policy number was corrected from being QA-SANA-002 to being QA-SANA-0002.

4.1.2.2. All references to Severn Trent Laboratories or STL have been replaced with TestAmerica, as a result of the integration of the STL and TestAmerica operations.

4.1.2.3. All other sections were modified only for clerical corrections.

Figure 1 - Method Blank Evaluation

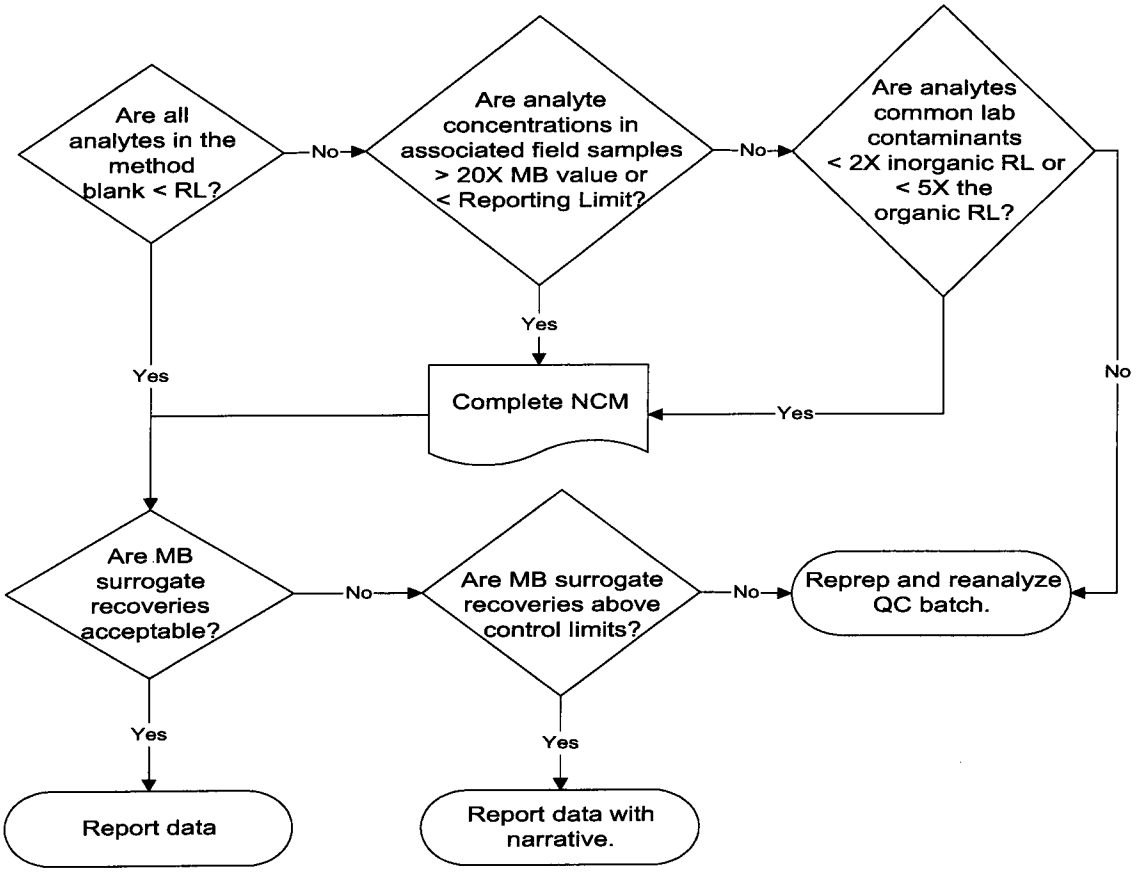


Figure 2 - LCS/LCSD Evaluation

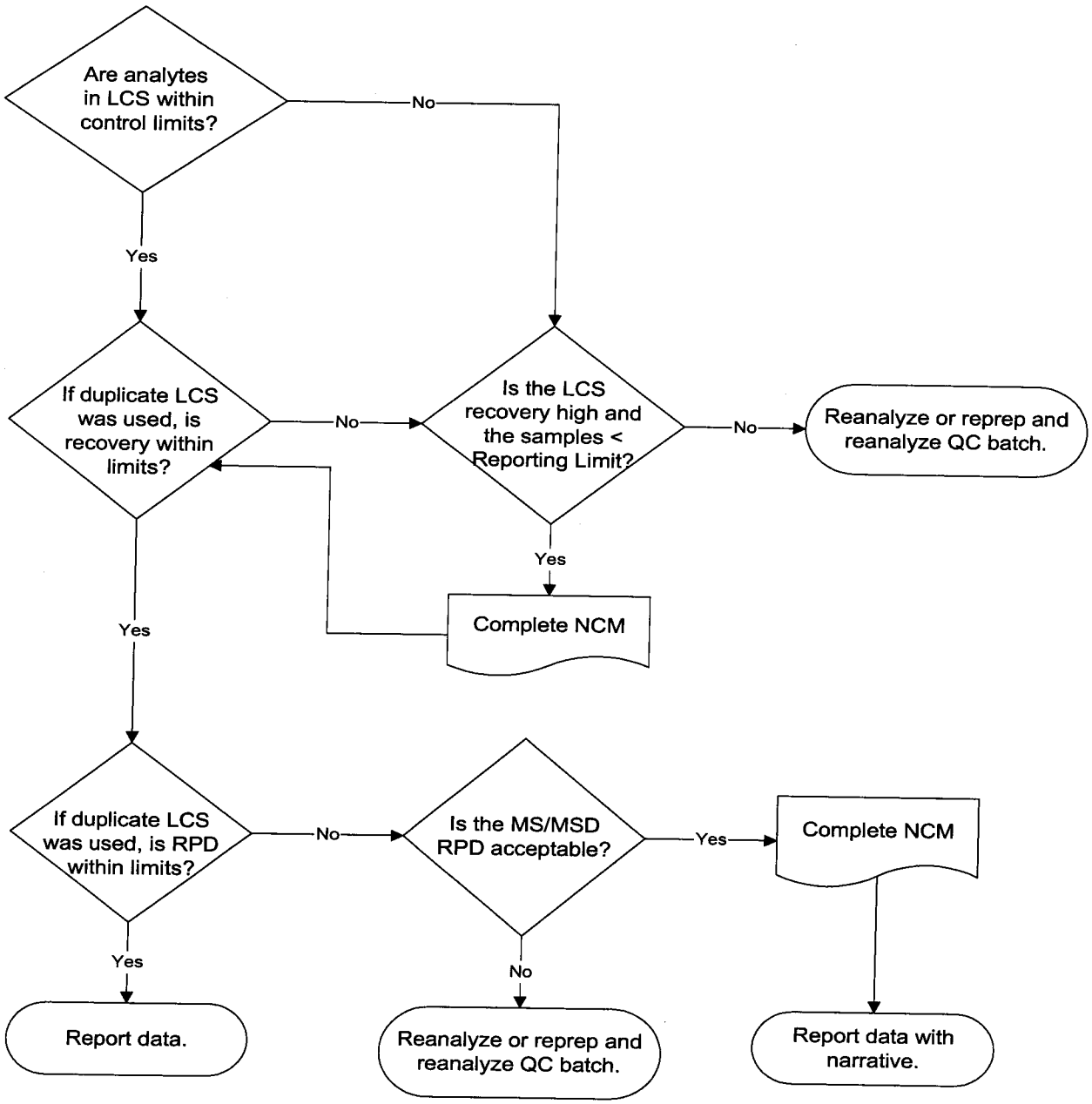


Figure 3 - Surrogate Evaluation

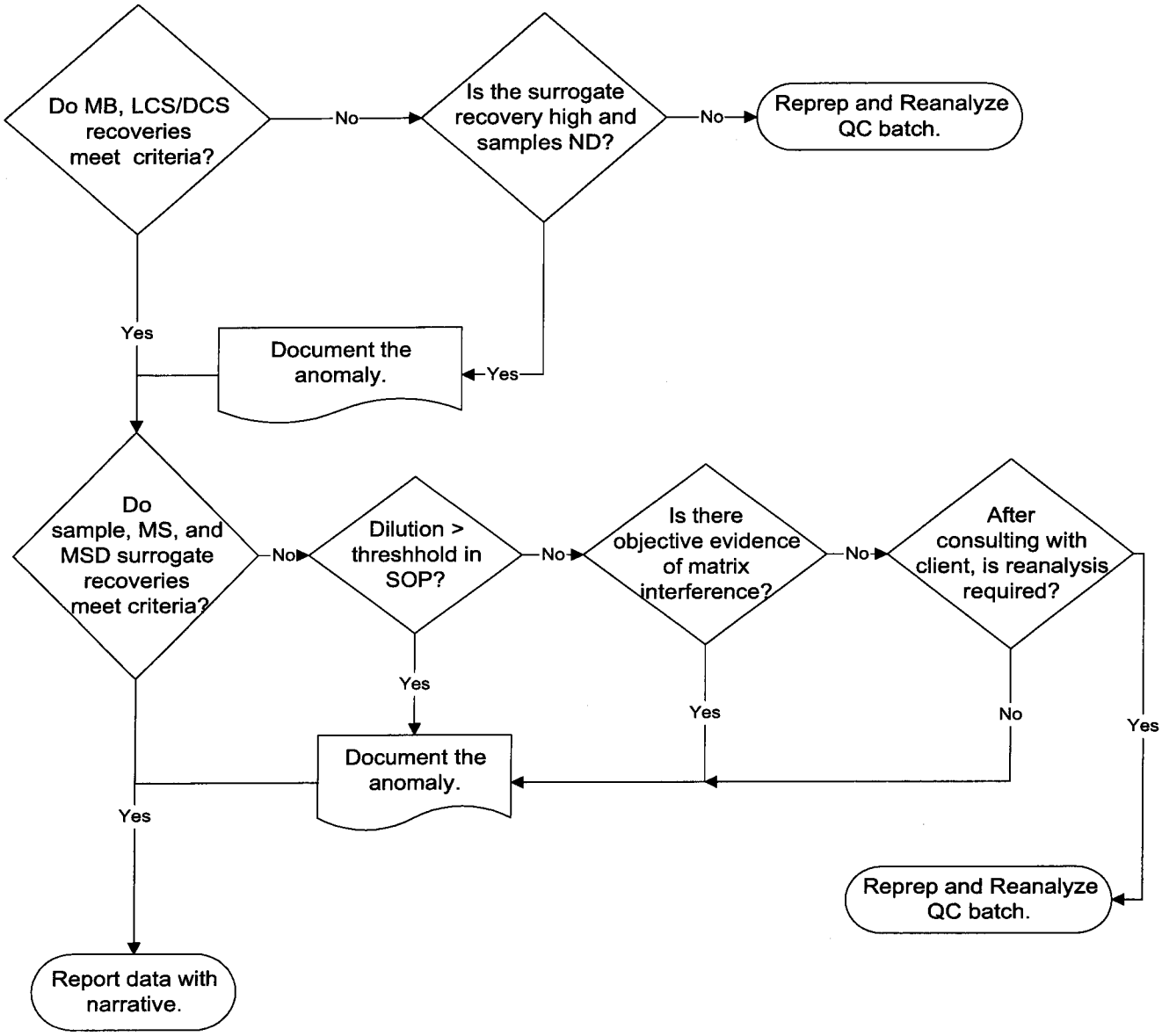
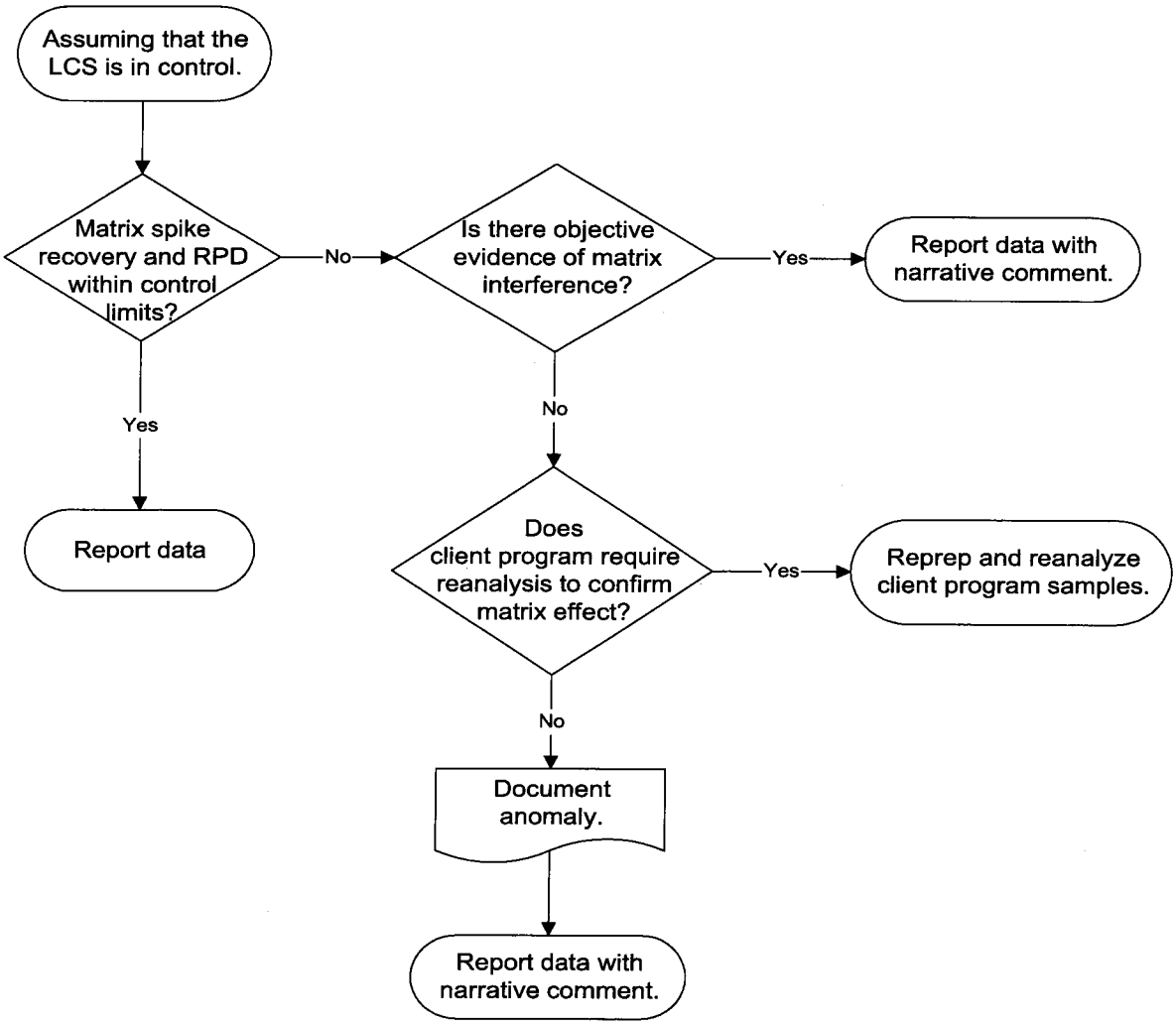


Figure 4 - Matrix Spike/Matrix Spike Duplicate Evaluation



Title: Corporate Document Control & Archiving

Approvals (Signature/Date):

10/24/07 Thomas R. Barr — Date Chairman/CEO Non-Analytical Division	10/18/07 Rachel Brydon Jannetta — Date CEO Analytical Division
10/10/07 Ilona Taunton — Date VP, QA and EHS	

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Controlled Source: Intranet **Facility Distribution No. _____**

1.0 PURPOSE

To identify Company Official Documents and describe the system of identification, collection, cataloguing, approval, maintenance and archiving (including retention) of those documents. This policy applies to the following operational areas: Technical and Client Services, Environmental Health & Safety, Finance & Procurement, Information Technology, Legal, Operations, Sales & Marketing, and Quality Assurance. This SOP is applicable to both the Analytical and Non-Analytical Divisions.

- To describe the personnel authorized to approve Official Documents.
- To describe the content, physical location and organization of Official Documents.
- To describe those documents that must be distributed and controlled.

The procedures for maintenance of records of distribution of controlled documents. This includes the location, review date and persons who have read and are responsible for the contents of an official document can be determined through the use of various tables described in this SOP.

2.0 SCOPE

Official company documents are Corporate Manuals, Policies, Procedures [e.g., Standard Operating Procedures (SOPs)], Work Instructions, White Papers and Training Modules. These documents are: a) authorized for use; b) controlled through the Corporate Quality Assurance Office; and c) must be distributed and controlled by each facility if they further distribute policies or procedures (beyond the intranet website).

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 **Control Binder** – The content and distribution to each binder is tracked by each facility's Quality Assurance Department for hardcopy distribution only.

4.2 **Controlled Copy** – Controlled copies of official documents are hardcopies or electronic copies that are maintained and updated by the Quality Assurance Department.

4.3 **Corporate Archives** – Original Official Documents retired from use, but retained for a specified time period for referential purposes.

4.4 **Corporate Manual** – A company-wide document describing a system of policies, procedures, organization and responsibilities for operating specific business functions. [e.g., Corporate EHS Manual].

4.5 **Document Control** – The system of identifying, cataloguing, approving, filing/storing and archiving of Official Documents.

4.6 **Policy** – A written statement or document identifying prudent conduct, a principle or a required activity to be pursued by the organization (e.g., the 'what' or 'what-not-to-do'). A Policy may be followed-up by a Procedure or a series of Procedures.

4.7 **Quality Record** – A document that defines the degree of effectiveness on how well a quality requirement is being met or how well the quality process is performing. It always documents what has occurred in the past.

4.8 **Standard Operating Procedure (SOP)** – A management directive that specifies the required activities and performance characteristics of a routine operation (e.g., the 'how-to-do-it'). A Procedure is the implementation of the Policy, if not otherwise described within the policy.

4.9 **Training Module** – Videos, presentations, handouts, tests/quizzes or other resources associated with company-required training.

4.10 **Uncontrolled Copy** – Uncontrolled copies are one-time distributions and updated revisions will not be distributed.

4.11 **Work Instruction** – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).

4.12 **White Paper** – A written response or summary of a topic that periodically requires some explanation of an industry or company issue. Allows for the capture of corporate knowledge and its preservation within the company. Although White Papers are official documents, they are not to be construed as company policy, procedure or as a quality record.

5.0 PROCEDURE

5.1 System of Identification

5.1.1 All Official Documents are identified and tracked on the Corporate Document Matrix. Document Numbers are assigned by the Corporate Quality Assurance Office.

5.1.2 Documents are associated with one of the following areas and are identified with a prefix for the Corporate Designation which is then associated to the Functional Group - Document Type - and finally a sequential number starting with -001 for each functional group.

Corporate Designation		Functional Group		Document Type		# Designation
CW	Company Wide	C	Customer Service	M	Corporate Manual	3-digit sequentially assigned number: 001, 002, etc..
CA	Corporate Analytical	E	Environmental Health & Safety (EHS)	P	Corporate Policy	
CNA	Corporate Non-Analytical	F	Finance & Procurement	S	Corporate Procedure (SOP)	
ESS	ESS	I	Information Technology	T	Corporate Training Manual	
IAQ	Indoor Air Quality	H	Human Resources	W	White Paper	
AIR	Air Operations	L	Legal	WI	Work Instructions	
QED	QED	O	Operations			
CDL	TA Drilling	T	Technology			
		S	Sales & Marketing			
		Q	Quality Assurance			

Following the table above, this Procedure under the Quality Assurance Functional Group, is identified as: **CW-Q-S-001**

Work Instructions use a similar scheme. The document type (WI) is followed by a 3-digit sequentially assigned number (beginning with -001) followed by the revision number. For example, the work instruction associated to the Corporate Document Matrix is: **CW-Q-WI–001.**

Manuals may have separate section identifiers such that they can be updated individually. In this case, an example document number for Section 1, Revision 1 of a manual would be **CW-E-M-001.1-Section 1**.

5.1.3 Facility System of Identification – Each Facility Location uses a 2-letter document prefix system to identify the documents facility-ownership (documents created or revised after issuance of this SOP). Refer to SOP No. CW-Q-S-002 for the facility system of identification.

5.2 **Collection of Information** - Official Documents may be authored by anyone within the organization. Such documents may be compiled by an individual, or as the result of the consensus of contributors. These documents are subject to approval as described in Section 5.3.

5.2.1 Cataloging of Information - The Corporate Document Matrix provides a listing of current documents. Information that is catalogued on each item is as follows:

- Corporate Designation
- Document Type
- Functional Group
- Director (of the Functional Group)
- Document Number
- Document Title
- Referenced In Documents:
- Revision Number
- Effective Date

NOTE: Refer to SOP CW-Q-S-002 on the format and content for 'Writing an SOP'.

5.3 Approval Process

5.3.1 All Official Documents must be submitted to the Corporate Quality Assurance Office to initiate the Approval Process. Corporate Manuals, Policies and Procedures are circulated for review and signature/date of approval.

5.3.2 All Corporate Manuals are approved by the Chief Executive Officer(s) (CEO); the Chief Operating Officers (COOs) or Business Unit President; and other Management members as appropriate.

5.3.3 All Corporate Policies and Procedures are approved by the COO's or Business Unit Presidents, Senior Management of a Functional Group as appropriate and the V.P. of Quality & EHS. When Corporate Policies and Procedures are relevant to all Functional Areas it may be more appropriate for the CEO(s) to sign-off on the SOP rather than the COO or Business Unit President.

5.3.4 White Papers are peer reviewed by a minimum of three (3) Quality Assurance (QA) Managers and at least one person outside of QA that is very familiar with the topic of the paper as defined by the Corporate Quality Assurance Office. The V.P. of Quality & EHS approves all White Papers prior to their posting on the Intranet.

5.3.5 Work Instructions are peer reviewed. Peer reviewers may include QA Managers, Directors of Functional Groups, V.P. of Quality & EHS, or any one person(s) directed by the Corporate Quality Assurance Office.

5.3.6 Training Modules are peer reviewed. Peer reviewers may include QA Managers, Directors of Functional Groups, V.P. of Quality & EHS, or any one person(s) directed by the Corporate Quality Assurance Office. A Directorial approval is granted prior to the posting of Training Modules on the Intranet.

5.3.7 The electronic version of an Official Document is archived by the Corporate Quality Assurance Office.

5.3.8 When electronic signatures are used for the approval process, a written approval or e-mail approval must be maintained to show approval to use the electronic signature.

5.4 **Document Maintenance** - The Corporate Quality Assurance Office maintains an electronic copy of all Corporate Documents.

5.4.1 The Corporate Document Matrix tracks the status of all aforementioned documents, and forms. The revision number, revision date and effective date are tracked to access the minimum of a 12-month review cycle of all Corporate Manuals, Policies and Procedures.

5.4.2 The Corporate Quality Assurance Specialist will initially review & update, as necessary, all documents for their annual review. The documents revision number will be updated to reflect this review cycle and the Revision History Section will summarize the specific section updates or note that no changes were necessary. This section provides a chronological account of the documents process development.

5.4.3 The electronic document is then circulated to all signatories for comments & approval. Once approved (e.g., E-mail confirmation), electronic signatures are imported into the cover page with the defined date of approval. The documents status is updated into the Corporate Document Matrix. Additionally, the Corporate Quality Assurance Office will E-mail all QA Managers and Directors of the associated functional group of an updated corporate document for implementation and use. All approved documents are posted on the intranet.

5.4.4 White Papers or Work Instructions, which are Official Documents but not Quality Records, are updated / revised as needed by the originator of the form or document.

5.5 **Document Archiving and Record Retention** - Official Corporate Documents are subject to the following retention policies:

Record Type	Retention Length
Customer Service	5 years
Environmental Health & Safety	5 years
Information Technology	5 years
Legal	5 years
Operations	5 years
Technology	5 years
Sales & Marketing	5 years
Quality	5 years

5.5.1 Outdated or retired documents will be electronically marked as **'Archived'** by the Corporate Quality Assurance Office and are electronically filed, with their email approvals, on the domain network. Both current and archived documents will be maintained in an electronic format only. [**Note:** Daily back-ups are performed as well as full-system backups on a weekly basis.]

5.5.2 An archived copy of each Official Document is retained for a period not less than that specified in the table above. The retention time begins at the time the document is retired or at the effective date of the new revision if a new revision is issued. The original signatory page or record of authorization of use of an electronic signature is kept in the archives. The archives are maintained by the Corporate Quality Assurance Office.

5.6 **Document Distribution** - The Corporate Quality Assurance Office controls all Official Documents by posting them in PDF format on the Intranet (Attachment 1). The footer on the cover page of each Corporate document (Figure 1) notes that the document is electronically controlled to the Intranet under the Documents tab (i.e., 'Controlled Source').

Figure 1.
Cover Page Footer for Corporate Documents

Controlled Source: Intranet	**Facility Distribution No. _____**

5.6.1 Facility Distribution of Corporate Documents – If the company facility prints & distributes corporate document(s) for internal use & adherence; a 'Facility Distribution No.' must be assigned as the associated internal tracking mechanism. These distributions are encouraged in an electronic format.

Each facility's Quality Assurance Manual & SOPs must detail their process for internal distribution of Corporate Documents as well as their administrative and technical procedures.

5.6.2 Release of Uncontrolled Corporate Documents

- It is the company preference that copies of Corporate Documents not be generated for use/review outside of the company. It is preferred that requested documents be reviewed on site only. It is suggested that a copy of the first page of the SOP be distributed when a copy of the document is being requested as proof that an approved document is available.

- Each facility will have document control procedures that describe their process for the distribution of Corporate Documents as well as their local documents.

6.0 RESPONSIBILITIES

6.1 Corporate Manual, Policy and Procedure approvals are as defined in Section 5.3.2.

6.2 The Corporate Quality Assurance Office is responsible for maintaining a system for assigning the document identifiers, maintaining and archiving the Official Company Documents as described in this SOP, and ensuring that the approved official documents are posted on the Intranet for implementation by all company facilities. The Corporate Quality Assurance Office will notify the Quality Assurance Managers via E-mail that a Corporate Manual, Policy, Procedure, White Paper or Work Instruction has been updated and approved.

6.3 The author of the Official Document is responsible for ensuring the accuracy of the information and submitting it to the Corporate Quality Assurance Office for assignment of the items defined in Section 5.1 before releasing any document for review.

6.4 The Non-Analytical Business Unit Managers (or their designee) and the QA Managers for the Analytical Businesses are responsible for the tracking and any non-intranet controlled distribution of Official Documents within or outside their facility.

7.0 REFERENCES / CROSS REFERENCES

Writing an SOP, Document No. CW-Q-S-002.

8.0 ATTACHMENTS

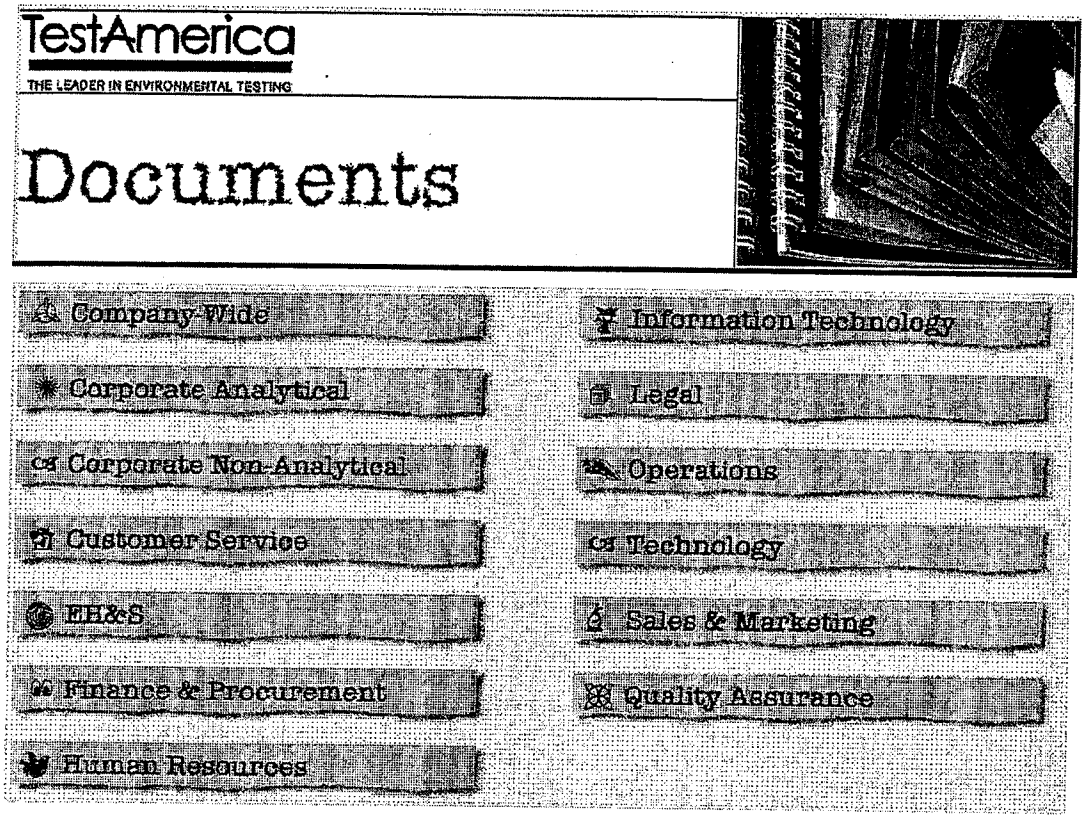
Attachment 1. Intranet Document Site.

9.0 REVISION HISTORY

- Revision 0, dated 11 October 2007
  - Initial Release.

Attachment 1.

Example: Intranet Document Site



TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Los Angeles

SOP No.: LA-QAS-001, Rev. 6
Effective Date: 11/06/2006

Title: STATISTICAL EVALUATION of QUALITY CONTROL DATA and DEVELOPMENT of CONTROL CHARTS

Approvals (Signature/Date):

9-14-2007	9/14/07
Maria Friedman Quality Assurance Manager	Date	Elizabeth Winger Laboratory Director	Date

This SOP was previously identified as SOP No. SANA-QA-0002.

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Facility Distribution No.: TestAmerica Los Angeles Intranet

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Los Angeles

FACILITY SOP ATTACHMENT

SOP ID: LA-QAS-001, Rev. 6	**CHANGE FORM ID:** CF1

SOP TITLE: Statiscal Evaluation of Quality Control Data and Development of Control Charts

REASON FOR ADDITION OR CHANGE: To document the change in ownership of the laboratory.

CHANGE OR ADDITION:

All references to "Severn Trent Laboratories, Inc." or "STL" in this SOP must now be referred to as "TestAmerica", in order to reflect the new ownership of the laboratory.

Likewise, this SOP has been renamed in accordance with TestAmerica Corporate QA policy, and a new cover page has been attached.

Prepared By: MARIA FRIEDMAN

***APPROVED BY:**

Quality Assurance Manager — Date: 9-27-2007

Laboratory Director — Date: 9/27/07

*Should be the same signature authorities of SOP being revised.

SEVERN TRENT STL

STL Los Angeles
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Santa Ana, CA 92705

Tel: 714 258 8610 Fax: 714 258 0921
www.stl-inc.com

STANDARD OPERATING PROCEDURE

TITLE: **STATISTICAL EVALUATION of QUALITY CONTROL DATA and DEVELOPMENT of CONTROL CHARTS**

(SUPERSEDES REV. 5)

Prepared by: William Daystrom

Reviewed by: 11-3-2006
Maria Friedman, Technical Specialist — Date

Approved by: 11-3-2006
Maria Friedman, Quality Assurance Manager — Date

Approved by: 11/03/2006
William Nash, Environmental Health & Safety Coordinator — Date

Approved by: 11-3-2006
Elizabeth Winger, Laboratory Director — Date

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1. SCOPE AND APPLICATION

1.1. This standard operating procedure (SOP) is applicable to the evaluation and establishment of QC acceptance criteria, and the long-term trend analysis of QC data using control charts or statistical tables at the STL Los Angeles laboratory.

1.2. The control chart is an effective tool for long-term trending because it records in real time the accuracy (bias) and precision of the appropriate parts of the measurement process. The control chart provides the means to demonstrate statistical control.

1.3. This procedure is to be enforced and followed by STL Los Angeles staff.

2. DEFINITIONS

2.1. Control Chart - A graphical QC tool to monitor method performance over time and to establish acceptance limits.

2.2. Relative Percent Difference (RPD) - a measure of intra-lab precision based on a duplicate sample or spike analyses.

2.3. Percent Recovery (%R) or Recovery - a measure of the accuracy (bias) of the measurement process based on a comparison of a measured value for a fortified (spiked) QC sample against the known spiked values.

2.4. Precision - a measure of mutual agreement (or variability) among individual measurements of the same property, usually under prescribed similar conditions.

2.5. Accuracy - the degree of agreement of a measurement (or an average of measurements of the same thing) with an accepted reference or true value. Accuracy is the measure of bias inherent in the system.

2.6. Bias - a systematic (consistent) error in test results. The difference between the population mean and the true or reference value, or as estimated from sample statistics; the difference between the sample average and the reference value.

2.7. X-chart - a control chart that plots a single measurement of a property (e.g., percent recovery) of quality control samples over time. The chart consists of a single line that is the mean of the statistic, warning limits at ± two standard deviations, and control limits at ± 3 sigma.

2.8. Assignable cause - a known reason for an outlying result (e.g., no spike added).

2.9. Duplicate - a second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

2.10. Laboratory Control Sample (LCS):

2.10.1. Organics: A LCS is a volume of deionized laboratory water for aqueous samples or a suitable solid material (e.g., clean sand) for soil/sediment samples which is spiked with compounds of interest and subjected to the entire analytical procedure in order to estimate the accuracy of the method via percent spike recovery.

2.10.2. Inorganics: A well-characterized, clean liquid or solid matrix sample that is prepared, digested or extracted along with each batch of samples.

3. RESPONSIBILITIES

3.1. Analyst

3.1.1. On a batch-by-batch basis, monitor analytical performance using QC samples (e.g., method blank, LCS, and MS/MSD) and identify any random or systematic out-of-control situation. This process does not require the generation of control charts.

3.1.2. Investigate the causes of out-of-control events as they occur.

3.1.3. Report these events to the Supervisor.

3.1.4. Notify the appropriate Project Manager as soon as an out-of-control QC excursion occurs that affect client samples.

3.1.5. Document any out-of-control excursions via Nonconformance process.

3.2. Group Leader/Supervisor

3.2.1. Respond to all out-of-control conditions. Implement adequate corrective measures to remedy deficiencies.

3.2.2. Communicate any systematic trends in the analytical process to the QA staff for resolution.

3.3. QA Personnel

3.3.1. Review control charts to detect any systematic trends in routine analytical procedures.

3.3.2. Archive control charts and statistically derived QC acceptance data.

3.3.3. Each year or as necessary, review and publish statistically-derived QC acceptance criteria based on historical performance data.

3.3.4. Oversee the incorporation of updated QC limits into LIMS.

3.4. Project Manager

3.4.1. Notify clients of updated QC data.

3.4.2. Incorporate updated limits into project-specific QAPPs.

4. SAFETY

4.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Lab Specific Addendum to the CSM, and this document.

4.2. Specific Safety Concerns and Requirements

4.2.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in the laboratory area, appropriate personal protective equipment and precautions must be utilized.

5. PROCEDURE

5.1. Evaluation and Empirical QC Acceptance Limits

5.1.1. The assessment of QC sample data shall be performed by comparing precision and accuracy results against control limits. As defined in the following subsections, the control limits used for this comparison shall be either in-house (statistically generated using historical data) control limits or published limits from methods.

5.1.2. In-house limits for all QC data must be evaluated and redetermined annually, and compared to those limits published in the methods for applicable matrices. Method limits will be employed until sufficient QC data are acquired. A minimum of 20 data points can be used to establish in-house limits based on historical performance data for each major method. If the laboratory does not have 20 data points, the method limits will be used until sufficient data are generated.

5.1.3. A new evaluation of control limits on a set of 20 data points may be warranted by a change in the analytical procedure.

5.1.4. Control limits shall be generated for each matrix (i.e., aqueous, solid, and air) for each method using data from at least 20 data points.

5.1.5. In-house control limits shall be established for the following samples:

5.1.5.1. LCS spike recoveries for target analytes.

5.1.5.2. Surrogate spike recoveries for organic analyses only in all sample types (client, LCS/DCS, MS/MSD, MB) collectively.

5.1.6. Control limits for MS/MSDs are established using the historical performance data generated based on LCSs (see section 5.1.5.1). If the recoveries in MS/MSD exceed the control windows, matrix effect has been demonstrated.

5.1.7. Control limits for MS/MSD surrogates are established using the limits calculated from all samples types collectively (see section 5.1.5.2).

5.1.8. During the routine review of control limits, if the newly generated QC limits are comparable to those established previously, the existing limits will be retained and no updates will be required. This will be clearly documented and communicated by the QA staff.

5.1.9. The calculations used to generate the control limits for accuracy (%R) are described in the following subsections.

5.1.9.1. The %R is defined as the observed concentration in LCS divided by the theoretical concentration of the spike or LCS, times 100:

$$\%R = \frac{Found}{True} \times 100$$

5.1.9.2. When the %R is obtained for at least 20 LCSs , the mean percent recovery and standard deviation are calculated using the following formulas:

$$\overline{\%R} = \frac{\sum_{i=1}^{n} \%R_i}{n} \qquad S_R = \sqrt{\frac{\sum_{i=1}^{n} \left(\%R_i - \overline{\%R}\right)^2}{n-1}}$$

where: %R = the mean percent recovery
%R I = the percent recovery of an LCS
n = the number of data points
s = the standard deviation of the data set of % recoveries

5.1.9.3. The warning (95% or 2-sigma) and control limits (99% or 3-sigma) are then calculated from the following equations:

5.1.9.3.1. Upper Control Limit = %R + 3 s

5.1.9.3.2. Lower Control Limit = %R - 3 s

5.1.9.3.3. Upper Warning Limit = %R + 2 s

5.1.9.3.4. Lower Warning Limit = %R - 2 s

5.1.10. Control limits will be recalculated after excluding the following points from the calculations:

5.1.10.1. Samples with values outside control limits due to assignable causes.

5.1.10.2. True outliers as defined in the Student t-test.

5.1.10.3. Outliers found using the Grubb's Outlier Test

5.2. Generation of Control Charts Using the Control Limits Program in Access

5.2.1. A control chart (X chart) is generated by plotting the LCS %R data in a graphical format as follows:

5.2.1.1. The average of the %R determinations for the original data set is established as the midpoint on the Y axis of the graph.

5.2.1.2. The upper and lower control limits are plotted as solid horizontal lines across the graph at their respective points on the Y axis.

5.2.1.3. The calculated %R of each spiked sample is plotted chronologically on the graph to determine whether the recovery is within the warning and control limits of the control chart.

5.2.2. As new LCS results are entered into LIMS, a real time control chart for a given period can be generated by section Supervisors to quickly evaluate trends as follows:

5.2.2.1. At the Main Menu of the Control Limits Program, click on the button labeled "On-Line Control Charts."

5.2.2.2. Select the desired QC Type (e.g., LCS/DCS, LCS/DCS Surrogates, All Surrogates) from the "QC Type" box.

5.2.2.2.1. For the proper calculation of surrogate limits, the option "All Surrogates" should be used.

5.2.2.3. Select an analytical date range to chart from the "Start Date" and "End Date" boxes. The default period is one month. Checkmark the "Limit Data Points" option to limit the chart to the most recent x data points (defined in the provided box).

5.2.2.4. Select a method to chart. Filter the list if necessary by using the "Group" option.

5.2.2.5. Select a QC Program to chart.

5.2.2.6. A list of LIMS Spike Lists for your chosen method/QC Program will be presented. Select a list to chart.

5.2.2.7. Click on the button labeled "Collect Data."

5.2.2.8. After a few moments, a control chart for the selected method, QC Program, and Spike List will appear on screen. If applicable, different constituents will appear on separate pages. The chart may be printed by right-clicking on the chart and selecting "Print."

5.2.3. Long-term Control charts can also be generated as follows:

5.2.3.1. At the Main Menu of the Control Limits Program, click on the button labeled "QA Access."

5.2.3.2. Select the desired QC Type (e.g., LCS/DCS, LCS/DCS Surrogates, All Surrogates) from the "QC Type" box.

5.2.3.2.1. For the calculation of surrogate limits, the option "All Surrogates" should be used.

5.2.3.3. Select an analytical date range to chart from the "Start Date" and "End Date" boxes. The default is one month. Checkmark the "Limit Data Points" option if desired to limit the chart to the most recent x data points (defined in the provided box).

5.2.3.4. Select a Spike List to chart.

5.2.3.5. Click on the button labeled "Collect Data."

5.2.3.6. When the query operation has finished, a message will appear indicating that the Grubbs Test has been applied to the data to screen for outliers. Clicking on "OK" will proceed to a form listing the constituents, existing and calculated control limits, and data points. An example is provided in Appendix A.

5.2.3.7. Data points that were determined to be outliers by the Grubbs Test will be so marked by a "Yes" in the data point's "Rejected?" column.

5.2.3.8. Control charts for the selected Spike List can be generated by clicking on the button labeled "Control Charts Rpt." An example of the output is provided in Appendix B.

5.2.3.9. A statistical report for the selected Spike List can be generated by clicking on the button labeled "Control Limits Rpt." An example of the output is provided in Appendix C.

5.2.4. The following information must be present on the control charts or in an associated statistical table:

5.2.4.1. Quality Control Type (i.e., LCS, Surrogate)

5.2.4.2. Constituent Name

5.2.4.3. Analytical and Preparation Methods

5.2.4.4. Date of Analysis

5.2.4.5. Matrix

5.2.4.6. LIMS Spike List ID

5.2.4.7. Statistical Calculations

5.2.4.8. Range of dates covering the period to be charted

5.3. Evaluation of Control Charts

5.3.1. The laboratory generated limits must be as stringent as and no wider than the method limits to evaluate QC samples, unless special analytical circumstances dictate that wider limits are more appropriate.

5.3.2. Criteria for an Out-of-Control Condition

5.3.2.1. The causes for a shift or a trend in control charts could result from:

5.3.2.1.1. incorrect preparation of a standard or a reagent

5.3.2.1.2. sample contamination

5.3.2.1.3. improper storage or preservation

5.3.2.1.4. incorrect instrument calibration

5.3.2.1.5. poor analytical technique

5.3.2.1.6. deviation from the analytical method

5.3.2.2. A measurement process for a particular analyte may be considered out of statistical control when one of the following conditions occurs:

5.3.2.2.1. a single point outside 3-sigma control limits

5.3.2.2.2. a series of eight successive points on the same side of the central line

5.3.2.2.3. a series of six consecutive points, such that each point is larger (smaller) than its immediate predecessor

5.3.2.2.4. a cyclic pattern of control values

5.3.2.3. When an out-of-statistical-control condition is identified, the data must be evaluated thoroughly to identify the most appropriate corrective action to be implemented. The problem and its solution may be documented through a Nonconformance Memo as appropriate. Exceeding warning limits will only require a close observation of the measurement system. In reviewing control charts, any significant changes in key analysts, instrumentation, standard reference materials, or processes must be kept in mind to explain potential out-of-control situations.

5.3.3. The form included in **Appendix D** can be used to document the trend analysis of control charts.

6. REFERENCES

6.1. Laboratory Quality Manual, current revision.

6.2. HAZWRAP Requirements for Quality Control of Analytical Data DOE/HWP-65/R2, September, 1996 or current revision.

6.3. Test Methods for Evaluating Solid Waste, Third Edition, SW-846, US EPA, Update III, 1996.

6.4. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EMSL, US EPA, March 1977.

6.5. Traqar Control Limits and Charts System software, version 4.

7. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

APPENDIX A

AN EXAMPLE CONTROL LIMIT EVALUATION FORM PRODUCED USING CONTROL CHART PROGRAM

Control Limits and Charts System - [Calc_Limits]

Control Limits Review Matrix: LCS/DCS

Constituent	Matrix	N	Mean	Std.Dev.	QuantIMS LCL	QuantIMS UCL	QuantIMS RPD	Initial Calculated LCL	Initial Calculated UCL	Initial Calculated RPD	Final Calculated LCL	Final Calculated UCL	Final Calculated RPD	UP?
1,1-Dichloroethene	[illegible]	[illegible]	[illegible]	[illegible]	70	130	25	[illegible]	[illegible]	[illegible]	76	126		
Benzene	[illegible]	[illegible]	[illegible]	[illegible]	75	120	25	[illegible]	[illegible]	[illegible]	81	119		
Chlorobenzene	[illegible]	[illegible]	[illegible]	[illegible]	80	120	25	[illegible]	[illegible]	[illegible]	83	118		
Toluene	[illegible]	[illegible]	[illegible]	[illegible]	80	120	25	[illegible]	[illegible]	[illegible]	78	120		
Trichloroethene	[illegible]	[illegible]	[illegible]	[illegible]	75	130	25	[illegible]	[illegible]	[illegible]	82	133		

1,1-Dichloroethene

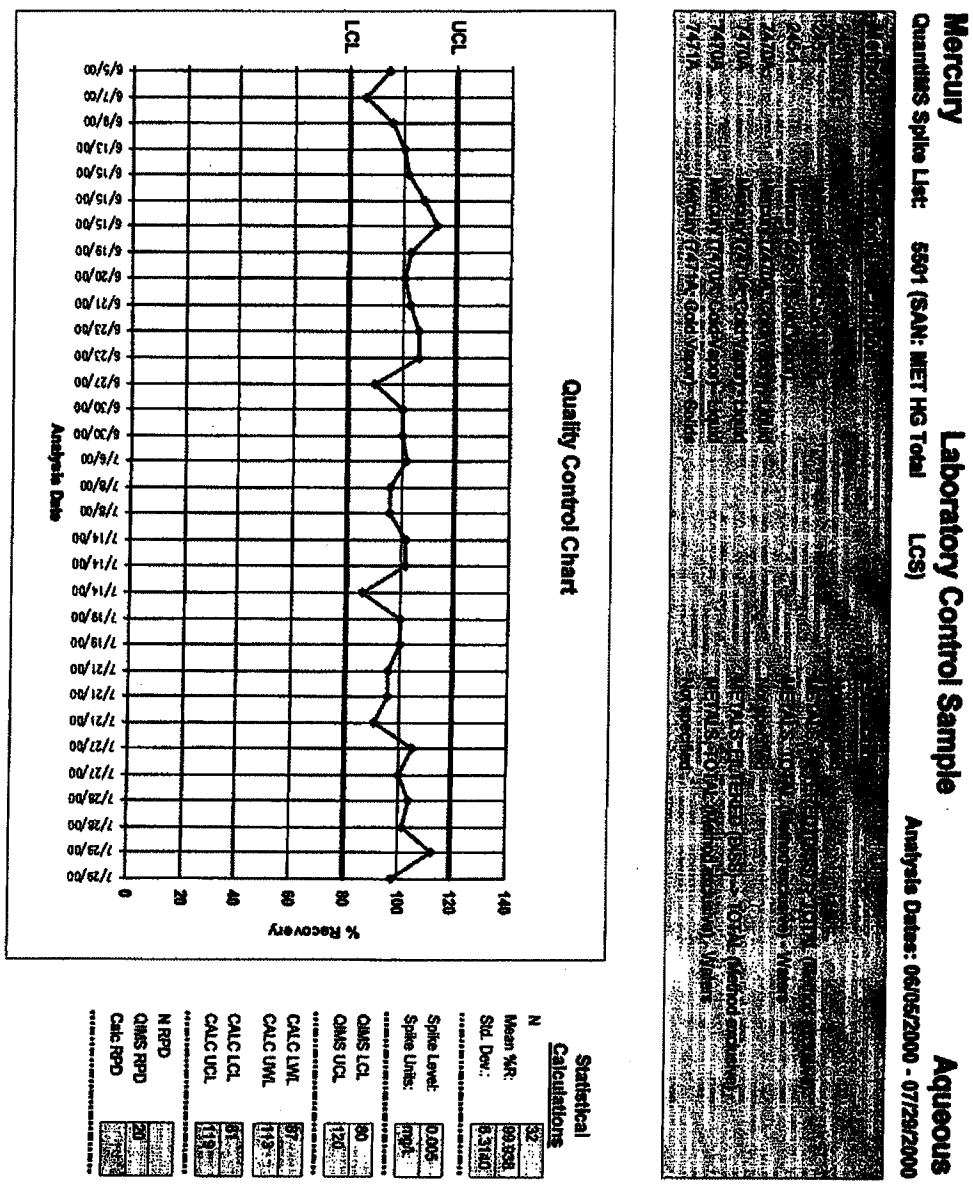
Sample ID	Workorder	Date	Recovery	RPD	Flagged
E0H150000264C	DHVE8102	20000814	76	0	No
E0F080000392C	DE9G6102	20000606	80	0	No
E0H040000539C	DHEP5102	20000804	80	0	No
E0F080000412C	DEE46102	20000607	86	0	No
E0F070000433C	DEC41102	20000606	87	0	No
E0F090000239C	DEF5V102	20000608	87	0	No
E0F190000533C	DF063102	20000619	87	0	No
E0F060000446C	DE95P102	20000606	88	0	No
E0H110000397C	DHQ2X102	20000810	89	0	No

Record: 1 of 25 (Filtered)

Form View

APPENDIX B

AN EXAMPLE CONTROL CHART REPORT GENERATED USING CONTROL CHART PROGRAM



APPENDIX C

PROGRAM GENERATED CONTROL CHART STATISTICAL REPORT

Control Limit Summary

Laboratory Control Sample

Los Angeles (s/w)

Spike List: 5501: SAN: MET HG Total LCS ***Analysis Dates: 06/05/2000 - 07/29/2000***

Aqueous

Constituent	Spike Level	Units	N	Mean	Std. Dev.	QuantIMS LCL	QuantIMS UCL	QuantIMS RPD	Calculated LCL	Calculated UCL	Calculated RPD
Mercury	0.005	mg/L	32	99.94	6.31	80	120	20	[illegible]	[illegible]	[illegible]

Solid

Constituent	Spike Level	Units	N	Mean	Std. Dev.	QuantIMS LCL	QuantIMS UCL	QuantIMS RPD	Calculated LCL	Calculated UCL	Calculated RPD
Mercury	0.833	mg/kg	13	96.54	3.50	80	120	20	[illegible]	[illegible]	[illegible]

Report generated: 8/17/00 1:04:45 PM ***Page 1 of 1***

Query request range: 6/1/00 - 8/1/00

APPENDIX D

CONTROL CHART TREND ANALYSES

SEVERN TRENT SERVICES

STL Los Angeles

CONTROL CHART TREND ANALYSES

Out-Of-Control Conditions

- ☐ Single point out of 3-sigma (σ) control limits
- ☐ A *cyclical* pattern of control values
- ☐ Other (*See Below*)
- ☐ Series of ***eight*** successive points on the same side of the central line
- ☐ Series of ***six*** successive points are such that each point is larger (or smaller) than its immediate predecessor

Description of Out-of-Control Condition:

Originator's Signature: **Date:**

Corrective Action Investigation
(To Be Completed & Reviewed by OPS Management)

Supervisor/Manager Signature:_____ **Date:**_____

Quality Assurance Review

QA Signature:_____ **Date:**_____

- **Is Corrective Action Satisfactory?** ☐ **Yes** ☐ **No**
- **Is an NCM Required?** ☐ **Yes** ☐ **No**
- **Corrective Action Verified By:**_____ **Date:**

8. SOP REVISION HISTORY

8.1. This section has been added beginning with revision 6. Prior revisions are documented in the QA files.

8.2. Changes to revision 5 implemented in revision 6:

8.2.1. Surrogate control limits are to be calculated using all sample types (client, LCS/DCS, MS/MSD, MB, and duplicates). Previously, surrogate limits were calculated using LCS samples only.

8.2.2. Section 4.1.6, regarding control limits for CLP projects, removed.

8.2.3. Sections were modified for clerical corrections.

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Sample Handling and Receiving for Asbestos Analysis

Environmental Microbiology Laboratory

Document Number:100202	Origin Date: 05/26/05	Revision Number: 1.1	Revision Date: N/A

Introduction

1. The following procedure enlists the safety requirements and the process for handling and receiving Asbestos samples.
2. Asbestos is known to cause asbestosis, mesothelioma, lung cancer; and cancers of the esophagus, stomach, colon and rectum and so proper protection and precautionary methods as stated in this document must be followed at all times while handling the asbestos samples.

References

1. Better protection against asbestos in the workplace, U.S. department of Labor, Fact Sheet No. OSHA 92-06.
2. NIOSH website http://www.cdc.gov/niosh/homepage.html
3. OSHA website http://www.osha.gov

Definitions

1. Asbestos: A family of naturally occurring minerals, found in serpentinite and other metamorphic rock.
2. Turn Around Time (TAT): The span of time in which the laboratory completes the analysis of a project, or portion of the project, from sample receipt to reporting of data.
3. Chain of Custody (COC): A legal record documenting the custody of the samples. A form completed by the client describing in detail all of the pertinent information required by the laboratory for analysis and reporting of the samples.
4. PACM: "Presumed Asbestos Containing Material". All bulk samples received in the laboratory for any type of analysis may or may not contain asbestos but must always be presumed to contain asbestos and so should be designated as presumed asbestos containing material.

Materials and media

1. Environmental samples
2. EML Project bins
3. Indelible blue pen and marker
4. Hand gloves
5. 70% Isopropyl alcohol and kimwipes
6. Bio-safety cabinet or hood attached with HEPA filters
7. Forceps

8. Scissors/box cutter
9. Knife/Blade
10. Half face air-purifying respirator
11. Ziploc bags (small, medium and large)

Procedures

1. The following practices must be strictly followed at all times when receiving and handling asbestos samples in the laboratory:
    1. When a shipment arrives count all of the packages to ensure that the number of packages received equals the number stated on the delivery drivers list.
    2. At all times while handling the samples proper protective clothing must be worn, as deemed necessary. The personal opening the packages shall wear a lab coat, and proper hand gloves, if needed.
    3. Open the package/box containing the samples carefully using a scissor/knife/box cutter or blade. Remove the samples and the chain of custody (COC) from the package and place in the appropriate colored bin depending on the type of turnaround (TAT) requested in the chain of custody (Use a red bin for a Same-Day Rush or Weekend/Holiday Rush, yellow bin for a Next Day, blue bin for Standard).
    4. On opening the package if it is observed that the samples received have not been properly sent in a sealed bag or container, then place the package in the bio-safety cabinet/hood and then remove, place and seal the sample carefully using a clean Ziploc bag.
    5. After all the samples have been appropriately contained/sealed and placed in the bin, then the sample can be logged in the database.
    6. Sample Acceptance/Rejection Policy:
        1. If it is observed that the bulk samples have not been properly contained separately and if it is suspected that the samples might have been cross-contaminated, then call the client and inform him about the situation and politely refuse to accept the sample for analysis.
        2. If the air and bulk samples have been sent together and there are chances that the debris from the bulk sample might have entered the air sample cassette and cross-contaminated the air sample than then call the client and inform him about the situation and politely refuse to accept the sample for analysis.
        3. If the air sample cassettes received for analysis is broken and damaged and the filter has been crushed then call the client and inform him about the situation and politely refuse to accept the sample for analysis.
        4. If the sample received is too small and a proper estimation cannot be performed then call and inform the client about the situation and request to obtain a larger sample size. If larger sample size cant be obtained and it is judged that the sample received is not sufficient to perform a proper analysis then politely refuse to accept the sample for analysis.
        5. If the type of sample received cannot be analyzed by the laboratory then call and inform the client about the situation and politely refuse to accept the sample for analysis.
        6. During any other situations if it is observed that the samples received may have been contaminated or damaged then make the judgement and call the client to discuss the situation and then determine the whether the samples may have been compromised.
        7. If at any time the client insists on performing the analysis for samples presumed to be contaminated or damaged, then leave a project log for the project indicating the request. This must then be transcribed in the report indicating the possibility of cross contamination.
    7. Further follow the procedures stated in the Environmental Microbiology Laboratory Document Number 100032 (Sample Receiving) and Document Number 100022 (Sample Log-In) for additional checking and log in of samples.
    8. After the samples have been logged in accordance to as stated above, then place all the

samples requiring asbestos analysis along with their COC into the bio-safety cabinet/hood for further prepping and analysis.

Calculations

Not Applicable.

Safety

1. Inspect and wear appropriate protective equipment/clothing as procedure dictates and when necessary to avoid exposure.
2. Personal protective equipment/clothing should not be your primary safety guard but it should be your last line of defense. Being vigilant and careful should be your first line of defense.
3. Do not remove any PACM from the contained cover/box at anytime when the sample is not within the bio-safety cabinet.
4. Wash all areas of exposed skin prior to leaving the laboratory.
5. Assume all bulk samples received in the laboratory for analysis to have asbestos and so handle them accordingly.
6. Always remain vigilant to any unsafe practices and conditions in the laboratory and immediately report such practices and/or conditions to the laboratory manager.

Reporting

1. Not Applicable.

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Work Out of Specification and Continuous Improvement

Environmental Microbiology Laboratory

Document Number:100000	Origin Date: 09/19/01	Revision Number: 1.6	Revision Date: 03/05/07

Introduction

1. EMLab has two protocols for addressing work or processes that fall outside of the standards set by the Standard Operating Procedures or the Quality Control Systems and another protocol for preventing potential errors. The first two are Work Out of Specification and Corrective Action and the last is Continuous Improvement:
   A. Work Out of Specification (WOOS)-Work Out of Specification is always the first step towards addressing issues that fall out of the normal range of expectation. Any work or processes that fall outside of the expected range of the internal Quality Control or are deemed by the employee as unusual and requiring correction are considered Works Out of Specification. Work Out of Specifications are reported as a Bug in a QA-zilla system. Employees are expected to report the incident by entering a Bug in the QA-zilla system and then inform their supervisor or manager. Employees are required to complete the EMLab ID, Summary, and Description sections in QA-zilla. All Corrective Actions are preceded by the Work Out of Specification Process. The priority of a Bug will be determined by the QA department.
      I. Any Work Out of Specification that is assigned Priority 1 must be immediately brought to the attention of the manager or supervisor of the department were the error occured, and in instances where the WOOS is highly significant, a Root Cause Analysis team is assembled. If the WOOS is highly significant, then work is immediately stopped and corrective actions are implemented before work resumes.
      II. The Laboratory Manager, the Quality Assurance Manager, and other senior level manager have the authority to stop or restart work.
   B. Corrective Action-Whenever a Work Out of Specification event is rated as significant by the managers and has possibly compromised the results of other tests and/or clients or a client contacts the laboratory with a significant concern, then the events described in the Corrective Action protocol (SOP Document Number 100001) are initiated. The steps involved in the Corrective Action process include Root Cause Analysis, Selection and Implementation of Corrective Actions, Monitoring of Corrective Actions, Auditing of Corrective Actions and Preventive Actions. Documentation must be kept for all for Corrective Actions and Corrective Action events are noted in the Quarterly Quality Assurance Audits.
   C. Continuous Improvement-Any work process or procedure that is judged by an employee as creating an undue amount of non-critical errors or could be improved to increase the quality or efficiency of the product or step should initiate the Continuous Improvement procedure. Continuous Improvement is a preventive action that captures potential sources of error or failures in the system before they become Corrective Actions. All Continuous Improvement

processes are entered in the QA-zilla system.

2. Examples of Work Out of Specification are temperatures out of the control limits for more than two consecutive days, temperatures that vary by more than two degrees from the expected, culture plates that have an uneven distribution of colonies or other growth pattern that indicates contamination, a recurring colony type on all culture plates regardless of the sampling location, sample labelling errors where true sample identification cannot be determined or is passed on to the next step without detection, contaminated blanks or sterility controls, repetitive logging or data entry errors, missed rushes, service related items, duplicate analyses that fall out of the specified confidence intervals, or any other process that falls outside of the boundaries set by the Quality Control systems, etc. A revision to or editing of reported data is always a Work Out of Specification that will initiate the Corrective Action procedure.
3. Work Out of Specification is not spelling corrections on log-in or reporting prior to reporting the results to the client, correcting data prior to submittal into the database or changing records on data entry forms, mislabelling of plates and slides that is correctable and the client information can be accurately traced, etc. If an employee has a question regarding whether a situation is a Work Out of Specification or not, the Quality Assurance Department can be contacted to determine the nature of the error.
4. Continuous Improvement is improving the quality of the processing of samples, identifying steps that could be streamlined or an excessive amount of errors that are not being measured or captured by the routine Quality Control steps, extra or repetitive steps that could be removed without decreasing the quality associated with the step or procedure or additional training by the employee. Employees should notify the Section Supervisor, Laboratory Manager or Quality Assurance Manager about any situation they believe could be a chance for Continuous Improvement.
5. Any Client Concerns are addressed by the processes detailed in SOP Document Number 100003.
6. The following document describes the laboratory processes that will be initiated when Work Out of Specification is encountered. The steps taken to control Work Out of Specification will include the definition of responsibilities and authorities for the management of the processes, along with an evaluation of the significance of the event, corrective actions taken, the chances for reoccurrence of the event and, when applicable, policies for notifying the client.
7. All information gathered during Work Out of Specification or Continuous Improvement events must be recorded into the QA-Zilla System. Follow the instructions in the "Work Out of Specification, Client Contacts, Continuous Improvements, and Corrective Actions using the the QA-Zilla System" (Document Number 100169).

Definitions

Work Out of Specification (WOOS)	Work or processes that fall outside of the specifications of the internal Quality Control or deemed by the personnel performing the task as not meeting the requirements of normal operation
Corrective Action (CA)	A Corrective Action is a process that addresses a Work Out of Specification event that is rated as significant by the management team and requires a change to the way a process or the work is performed or an adaptation to the Quality Control System associated with the step or process
Change Control (CC)	The process by which changes are made to any controlled document that defines procedures, processes, products, etc. The process involves formally updating all associated documents and officially removing and archiving all the non-updated versions of the document
Root Cause Analysis (RCA)	A process used to identify the cause of a problem or error. The process involves identifying all of the portions of a problem and through the process of elimination, narrowing the field down until a cause can be agreed upon by consensus. (SOP Document Number 100019)

Quality Control (QC)	Technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of the end users. The aim is to provide quality that is satisfactory, adequate, dependable and economical

References

1. Laboratory Quality Assurance Programs Policies, American Industrial Hygiene Association, Section 2.7.10.8 Page 12, April 2001
2. General requirements for the competence of testing and calibration laboratories, International Organization of Standardization, Section 4.9 Page 7, 1999-12-15

Materials and Media

1. A Computer with internet access
2. A Labserve account and QA-zilla account
3. Work Out of Specification, Client Contacts, Continuous Improvements, and Corrective Actions using the QA-zilla System Standard Operating Procedure (Document Number 100169)
4. Resolving Client Concerns Standard Operating Procedure (Document Number 100003)
5. Root Cause Analysis Standard Operating Procedure (Document Number 100019)
6. Change Control Standard Operating Procedure (Document Number 100020)

Procedures

1. Work Out of Specification
   A. When a laboratory procedure falls outside of the specifications of the Quality Control system or is deemed by the employee as unusual and requires addressing is considered Work Out of Specification. Follow the steps outlined in the Work Out of Specification, Client Contacts, Continuous Improvements, and Corrective Actions using the QAzilla system SOP (Document Number 100169).
   B. If an employee identifies an error made by themselves or receives a client complaint, then that person should report the incidence by entering a bug in the QAzilla system. When a person identifies an error made by another employee, then the employee or department who made the error should be informed of the error and enter a bug in the QAzilla system.
   C. Any Work Out of Specification that is assigned Priority 1 must be immediately brought to the attention of the Quality Assurance department and/or Senior Management.
   D. If the problem is determined to be highly significant and has possibly compromised the results of other tests and/or clients or is related to the data reported to clients, then the steps outlined in the Corrective Actions SOP (Document Number 100001) must be taken. The actions to be considered must include, but not be limited to, the following: Halting of work, quarantining other associated work, withholding of reports, reassessing the QC procedures, and contacting the clients if reported results have been affected, until Corrective Actions have been identified and implemented.
   E. For routine Work Out of Specifications, the QA Manager and the Laboratory Manager and/or Section Supervisor will then determine the immediate actions that need to be taken. If the problem is determined not to be highly significant and identified before the results were released to the client, then Corrective Actions will be identified and implemented by the QA Manager and the Laboratory Manager and/or Section Supervisor.
   F. If the client is contacted the following procedures should be considered:
   G. Admit to the problem and apologize.
   H. Get a resolution and try to minimize the impact to the client.
   I. The Quality Assurance Manager will be responsible for ensuring that any Corrective Action is implemented and that Work Out of Specification issues are addressed and documented in the Quarterly Quality Assurance audit.
2. Continuous Improvement

   A. A situation or process that can potentially be improved or errors that can be avoided are considered Continuous Improvement.
   B. When situation or work process that can be improved or any situation that the employee feels can be changed to improve the quality of the work performed by the laboratory is encountered, the employee must initiate the Continuous Improvement process by entering a bug in QAzilla.
   C. Follow the steps outlined in the Work Out of Specification, Client Contacts, Continuous Improvements, and Corrective Actions using the QAzilla system SOP (Document Number 100169).
3. The Priority of the bug will be assigned by the QA department on a scale of one to five. The ratings will be based on the following criteria:
   I. Priority 1: Significant Impact on Interpretation. Integrity of data compromised.
   II. Priority 2: Possible Minor Impact on Interpretation. Integrity of data moderately compromised.
   III. Priority 3: No impact on interpretation. Data compromised.
   IV. Priority 4: No impact on interpretation. Data possibly compromised.
   V. Priority 5: No impact on data. No data integrity issue.

Calculations

1. Not Applicable

Safety

1. Not Applicable

Quality Control

1. The Quality Assurance Manager is responsible for ensuring that all WOOS's are investigated and Corrective Actions are identified and implemented.

Reporting

1. All Work Out of Specification Bugs reported will be archived in the QAzilla system.

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Quality Control for Asbestos Analysis

Environmental Microbiology Laboratory

Document Number:100201	Origin Date: 05/26/05	Revision Number: 1.2	Revision Date: 01/06/06

Introduction

1. The following document details the different quality controls for analysis, materials, reagents, microscopes and the workplace that must be performed in the laboratory.
2. Any information contained within this document is subject to change without notice.

References

1. Laboratory Quality Assurance Programs Policies, American Industrial Hygiene Association, Module 2, April 2005.
2. Standard Methods for the Examination of Water and Wastewater, American Public Health Association, Section 9020 B-8 Pages 9-9 and 9-10, 20th Edition, 1998.
3. Statistical Quality Control Handbook, Western Electric Co., Inc. (AT&T), Section B-3, Pages 12-16, 1956.
4. Guide for Quality Control on the Qualitative and Quantitative Analysis of Bulk Asbestos Samples: Version 1. Jennifer R. Verkouteren, David L. Deuwer. NISTIR 5951.

Definitions

1. Not applicable

Materials and media

1. Computer data file for statistical analysis (.xls)

Procedures

1. Reference Sample Analysis
   A. PLM - The Reference Sample Quality Control Analysis (PLM) is performed by each analyst on a monthly basis to evaluate the precision and accuracy of each analyst for fiber identification. The goal of performing Monthly Reference Sample Quality Control Analysis is for continuous improvement. The Quality Assurance department will randomly select a sample for each analyst from this collection of reference samples. Each analyst will analyze the assigned sample for each month, recording their results for the identifications. The identification by each analyst will be compared with the known standard by the Quality Assurance Department. Any analyst who has misidentified a fiber, will be asked to review the slide again to determine the source of error. If necessary, corrective actions will be

recorded as determined by the Laboratory Manager based on the nature of the error.

B. PCM - The Reference Sample Quality Control Analysis (PCM) is performed by each analyst on a daily basis to evaluate the precision and accuracy of each analyst for fiber identification. The goal of performing Daily Reference Sample Quality Control Analysis is for continuous improvement. The samples for the Daily Reference Sample Quality Control Analysis consist of reference permanent slides, each of which contains varying asbestos or non-asbestos fiber. The Quality Assurance department will randomly select a slide for each analyst from this collection of permanent slides. Each analyst will analyze the assigned slide for each day, recording their results for the identifications. The identification by each analyst will be compared with the known standard by the Quality Assurance Department. Any analyst who has misidentified a fiber, will be asked to review the slide again to determine the source of error. If necessary, corrective actions will be recorded as determined by the Laboratory Manager based on the nature of the error.

2. Proficiency Analytical Testing Participation
   A. The laboratory will participate in the external proficiency testing program conducted by AIHA or NVLAP.
   B. The goal of participating in an external proficiency testing program is for continuous improvement and to measure accuracy and precision of the laboratory.
   C. The samples received for proficiency testing will be analyzed individually by every analyst conducting asbestos analysis. If discrepancy in the fiber identification of the sample is noted then the analysts will be asked to review the sample again to determine the source of discrepancy. If necessary, corrective actions will be recorded as determined by the Laboratory Manager based on the nature of the discrepancy.
   D. The final identification with the group concensus will be submitted to the proficiency testing program.
3. Inter-Laboratory Round Robin Analytical Testing Participation
   A. The laboratory will participate in an external proficiency testing program for bulk sample asbestos analysis with other asbestos testing laboratories . The goal of participating in an external inter-laboratory proficiency testing program is for continuous improvement and to measure accuracy and precision amongst the cooperating laboratories.
   B. Data will be statistically analyzed and control limits will be determined for the analyses as stated earlier in the section of "Duplicate and Replicate Analysis".
   C. The QA department will chart the replicate counts and inform the Laboratory Manager of any identification or percentage count that have gone outside of the specified range. The Laboratory Manager will then be responsible for ensuring that the analysts recheck the identification or percentage count and determine the source of variation.
   D. If the errors are significant, Corrective Actions will be identified and implemented by the QA department and the laboratory manager.
4. Quality Control for Materials, Reagents and Microscopes
   A. Contamination Controls: The following contamination control must be done before and/or after asbestos examination,
      I. The sampling instruments (forceps, spatulas, cutters, etc) must be washed with water/alcohol and wiped dry with kim wipes before and after use. They must be frequently checked under the stereoscope to make sure they are free of asbestos fibers.
      II. The workbenches (including the area inside the HEPA filter hood), stereoscopes and microscopes must be wet wiped with paper towel or pre-moistened kimwipes.
      III. The glass slides and cover slips, must be checked for contamination before use. The refractive index (RI) liquids must be checked for contamination at least twice daily or whenever contamination is suspected. One drop each of 1.550, 1.605, and 1.680 RI liquids are placed in three different areas of a slide, followed by covering of the drops with cover slips. The three areas are then examined under a PLM to check for contamination. A log of the contamination check for the RI liquids conducted will be recorded by the analyst. The log sheets will be maintained and filed by the QA department.

IV. All contaminated disposal materials must be discarded. The cleaning and checking for contamination of non-disposable supplies, apparatus, implements, and lab benches must be repeated until the materials are found to be free of asbestos fibers and dirt.
V. A log for contamination checks conducted on the microscope and RI liquids will be entered by the analyst conducting the analysis. The log will be filed and maintained by the QA department.

B. Workplace Environment Contamination Control: The following contamination check for the workplace environment will be conducted monthly,
   I. At the beginning of the month, one clean glass slide must be exposed on top of each workbench and under each HEPA filter hood. At the end of each month, the slides must be collected and scanned for asbestos contamination under the PLM. If asbestos fibers are found, the contaminated area must be cleaned using the procedures as stated in the section "Contamination Controls".

C. Microscope Alignment
   I. The microscope must be checked for set up and aligned daily prior to use according to the procedure as stated in the document "Sample Preparation and Analysis for Asbestos and Other Fibers by Polarized Light Microscopy (PLM) (EPA Method 600/R-93/116) – Bulk Sample Analysis" SOP (Document Number 100204) and "Sample Preparation and Analysis for Asbestos and Other Fibers by PCM (NIOSH 7400) – Air Sample Analysis" SOP (Document Number 100203).
   II. A log for the microscope alignment will be entered by the analyst conducting the analysis. The log will be filed and maintained by the QA department.

D. Calibration Controls
   I. Temperature Control: The temperature of the workplace next to the location of the microscope will be monitored and recorded at least twice daily. A log of the temperature recorded will be maintained and filed by the QA department. If at any time the temperature is found to drastically vary from the regular room temperature of 23±2°C, then appropriate measures will be taken to restore the temperature back to the normal level.
   II. RI liquid calibration: Follow the procedures stated in the paper/document "Refractive Index Liquid Calibration Using Optical Glass Standards" by Shu-Chun Su filed as a hardcopy along with the protocols to calibrate the RI liquids 1.550, 1.605 and 1.680. These three RI liquids will be calibrated monthly. The other liquids that are rarely used in bulk asbestos identification need to be calibrated only prior to use. If the RI liquid values fall within an accuracy of ±0.004 then the RI liquids can be used for analysis, if not they should be discarded. A log of the calibration results will be filed and maintained by the QA department.

E. Asbestos Reference Materials
   I. Asbestos reference materials are obtained the National Institute of Standards and Technology.
   II. Intermediate checks will be performed annually to maintain confidence in the status of materials.
   III. Reference standards must be kept at room temperature in containers provided by the National Institue of Standards and Technology.

Calculations

1. Not Applicable

Safety

1. Follow the safety practices described in the General Laboratory Safety Procedures and Chemical Hygiene Plan.

Quality Control

1. Not applicable

Reporting

1. All Root Cause Analyses and Corrective Actions are documented in the QAzilla system.

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Document Control and Control of Records

Environmental Microbiology Laboratory

Document Number:100017	Origin Date: 10/01/01	Revision Number: 1.11	Revision Date: 03/07/07

Introduction

1. All documents that are part of the quality assurance system, either internally generated or external, shall be controlled. These documents include, but are not limited to, control charts, equipment manuals, training forms, personel forms, reference texts, MSDS manual, software, QC forms, reports, and SOP's.
2. It will be the duty of the QA Coordinator and Laboratory Director or their designates to approve all of the above for initial use and to review periodically.
3. Procedural and Quality Control documents are generated following the protocols outlined in the EML Methodology for Generating Standard Operating Procedures (Document Number 100024). All documents that are edited or revised must follow the protocol described in the Change Control Procedures SOP (Document Number 100020). All documents must first be cleared by the Quality Assurance Department before being generated. The Quality Assurance Manager will assign a Document Control Number and the document must be signed off by the President, Laboratory Manager, or other Managerial Designee, and Quality Assurance Manager before being put into use.
4. All document control numbers are maintained in an Excel file with restricted access. The Quality Assurance Manager, the Quality Assurance Coordinator, and the President have access to the file.
5. Standard Operating Procedures and other controlled documents must not be removed from the laboratory (unless designated by the management for release to customers) or shared with any one other than laboratory employees.

Definitions

Document Control	A system of protecting the integrity of processes and systems in the laboratory by uniquely identifying and monitoring the alteration to documents within the laboratory. All new documents, editions, or changes to documents must be signed off by the laboratory management
Change Control	The process by which changes are made to any controlled document that defines procedures, processes, products, etc. The process involves formally updating all associated documents and officially removing and archiving all the non-updated versions of the document
Document Archiving	A system of safely storing data that ensures that the records remain intact, reviewable, and retrievable for a specified period of time

References

1. General requirements for the competence of testing and calibration laboratories, International Organization of Standardization, Section 4.12 Page 8, 1999-12-15

Materials and Media

1. Change Control Procedures (SOP Document Number 100020)
2. Work Out of Specification, Client Contacts, Continuous Improvements, and Corrective Actions using the QA-zilla System SOP (Document Number 100169)
3. Methodology for Generating EML Standard Operating Procedures (SOPs) (Document Number 100024)
4. SOP Version Control System: Concurrent Versions System (CVS) (Document Number 100168)

Procedures

1. **Document Control**
   A. All documents associated with the processing of samples and other related activities in the laboratory are assigned Document Control Numbers. The specific categories for each type of document are described in the Introduction Section of this document.
   B. Each document is assigned a six digit number according to a designated categorical listing. The listings are as follows:
      I. 100000 Series-Standard Operating Procedures
      II. 200000 Series-Forms, Control Charts, Graphs
      III. 300000 Series-Policies
      IV. 400000 Series- Reference Texts, Scientific Journals, and other Technical References
      V. 500000 Series- Equipment Manuals
      VI. 600000 Series--Software
      VII. 700000 Series--MSDS's
      VIII. 800000 Series- Marketing Documents
      IX. 900000 Series- Media Formulations
   C. If a document is produced by the laboratory for a procedural and/or quality control system, then the Quality Assurance Manager must approve the document and assign a document control number. The document control number will then be entered into an Excel file that contains the records of all the documents produced by the laboratory.
   D. All Standard Operating Procedures must follow the processes outlined in the Methodology for Generating EML Standard Operating Procedures (SOP Document Number 100024). Each document must have an Origin Date and Revision Number assigned by the Quality Assurance Manager.
   E. After any document is completed, the President, Laboratory Manager or other Managerial Designee, and Quality Assurance Manager must sign off the document before it is implemented in the laboratory. All documents related to sample analysis must be signed off by the Laboratory Manager.
   F. Whenever a document is edited or revised, all copies of the older version of the document must be collected before the revised version is implemented, if printed versions are available. All versions viewed through the intranet with be automatically updated. All affected personnel must be notified of the change and briefed on the changes in the document. Employees will be notified of changes to documents through the QA-zilla system.
   G. All Change Control Numbers will be the Bug Number generated automatically by the QA-zilla system. Changes to any document must be recorded using the Change Control Procedure as outlined in Document Number 100020.
   H. Records are kept a minimum of three years by the laboratory. Hard copies of all non-electronically recoverable files are kept in filing cabinets until being placed in document storage boxes and stored at an archival facility. Computer records are backed up each weekday. The back-ups are kept in the Data Management Department in a fire-proof safe.

Monthly the data files are backed up and the tape is kept off site. Annual tests must performed to ensure that backed up data can be retrieved.

I. A listing of externally obtained documents such as equipment manuals and reference texts will be maintained and updated annually. Old versions or editions will be marked as such and taken out of circulation. Any changes to documents will be minor changes only and will be clearly marked, initialled, and dated. These changes can only be made by the QAC or Lab Manager or designate.

J. External Documents:

I. Reference Texts, etc.: A listing of books, journals, and other technical reference sources shall be made and cataloged by the above designation. The list shall be reviewed and updated periodically.

II. Equipment Manuals: All items of equipment should have a manual available for reference. A list of all equipment manuals shall be maintained using the above designation. When a new item of equipment is received, the manual should be given to the QAC for inclusion in the listing. The listing shall be reviewed and updated at least annually.

III. Software: All software used by computers at EML shall be referenced using the above designation. Any new software should be added to the listing by the IT. The listing shall be updated at least annually.

IV. MSDS's: A file of all MSDS sheets will be indexed as above and kept in a binder. The index shall be updated at least anually.

V. Media Formulations: A copy of the formulation label from each container of media shall be kept in a file and indexed using the above designation. The index of media formulations shall be updated at least annually.

2. **Control of Records**

A. Client Records:

I. Records must be meticulously maintained. All completed documents associated with a project must be filed together. The file must contain all documents that can not be reproduced electronically, including, but not limited to, the following documents:

a. Copies of clients checks sent in for payment

b. Hard copy or facsimile of all original data sheets that are not from a terminal entry, if applicable.

c. Any faxed or mailed correspondence from a client, especially if the correspondence is related to the changing of a analysis type and/or sample volume, etc.

II. Files are sorted by invoice date. Record the invoice date on the outside of the filing cabinet to facilitate finding records.

III. Store archived files on site as long as possible. When space becomes limited the files must be archived at a facility approved by the senior management or the quality assurance department. The invoice date ranges for the files must be clearly written in indelible ink on the outside of the archival boxes. If the date range for a set of files requires more than one box, then the alphabetical range for the files must also be included on the side of the archive box.

B. Laboratory records:

I. Laboratory notebooks will be kept with the lab managers.

II. All QC records including, temperature charts, daily microscope logs, equipement lists, preventive maintenance forms will be kept in QC binders.

C. Internal audit/management review records:

I. Internal audit forms and Management review records are kept in labeled folders and kept in filing cabinets in main and regional labs.

II. Internal audit forms and Management review records will be kept in QC binders in Microlabs.

D. Technical staff training/qualification records:

I. Technical staff training forms and qualification records will be kept in the following places:

a. A filing cabinet in QA Manager's office.
b. A filing cabinet in Human Resource Department.
c. A filing cabinet in the lab managers's office.

E. Equipment Manuals:
I. All laboraotry equipment manuals must be kept in QC binders or filing cabinets.

F. Forms and records are stored in alphabetical order.

3. **Numbering of Computer Files**
A. Printed copies of documents will be released for distribution by the Quality Assurance Manager or found in designated Standard Operating Procedures binders in the various sections of the laboratory or through the intranet.
B. All references to other related documents within a SOP or other controlled document will identify the document by the Document Control Number.
C. Equipment Preventive Maintenance Recording Forms are available from the QA department. The documents can be searched using one of the following methods: The Document Control Number, the two digit equipment code or the EML equipment ID tag number. The files are named using the following format: 200016_CC_ID200013, where the first six digits are the Document Control Number, the second alpha characters are the equipment code, and the last series of alpha and numeric identifiers are the EML ID tag numbers. Following is a list of the two letter equipment codes.
I. AB-Analytical Balance
II. AU-Autoclave
III. BH-Benchtop Hood
IV. CC-Colony Counter
V. CT-Cryo-Storage Tank
VI. ER-ELISA reader
VII. ET-Endotoxin Instrument
VIII. FO-Fiber Optic Illuminator
IX. BS-Biosafety Hood
X. HP-Hot Plate
XI. IN-Incubator
XII. MF-Membrane Filter Manifold
XIII. ML-Magnifying Lamp
XIV. MW-Microwave
XV. MS-Microscope
XVI. RF-Refrigerator
XVII. PP-Pippettor
XVIII. PS-Pouch Sealer
XIX. ST- Stomacher
XX. TH-Thermometer
XXI. VM-Vortex Mixer
XXII. WB-Water Bath

4. **Monitoring the revision of documents using the Concurrent Versions System (CVS)**
A. All documents are revision controlled by the CVS system. Any changes to documents must follow the protocols outlined in the SOP Version Control System: Concurrent Versions System (CVS) SOP (Document number 100168).
B. The CVS system keeps records of all changes to documents, but normal Change Control procedures must be followed (Document Number 100020) to ensure that proper management sign-off to document changes is recorded. A hard copy of the most recent signed revision of all controlled documents must be stored by the QA department.
C. Whenever a change to a procedure is identified by a Corrective Action it must be recorded in the appropriate document following normal Change Control procedures.
D. All current versions of documents are posted on the intranet. Previous versions of documents are archived within the CVS system.

Calculations

1. Not Applicable

Safety

1. Not Applicable

Quality Control

1. All signed Controlled Documents are stored in a locking file cabinet that is accessible only to the Owner, President, Laboratory Manager, and the Quality Assurance Manager.
2. Once a year the stored data files must be tested to ensure that the data stored can be easily retrieved. The test is part of the QA audit process.
3. (10/09/03) The WOOS, Corrective Actions, and Client Concern forms and numbers were obsoleted because the system for tracking the quality functions has been transferred to the QA-zilla system which generates it's own tracking numbers. For more information on the QA-zilla system, refer to the "Work Out of Specification, Client Contacts, Continuous Improvements, and Corrective Actions using the QA-zilla System" SOP (Document Number 100169).
4. (5/21/04) The Change Control forms and numbers have been obsoleted because the tracking of change controls will now take place in the QA-zilla system. For more information refer to the Change Control Procedure SOP (Document Number 100020)

Reporting

1. All reviews of the controlled documents will be summarized in the annual and/or quarterly QA audits.

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TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. CORP-MS-0002NC, Rev. 2.5
Effective Date: 04/03/07
Cover Page

Title: Determination of Volatile Organics by GC/MS
[Methods: 8260B, 8260A and 624 (Note: Update II and Update III are in one SOP)]

Approvals (Signature/Date):

Technology Specialist	12/14/07 Date	Health & Safety Coordinator	12-14-07 Date
Quality Assurance Manager	Date	Laboratory Director	12/14/07 Date
Technical Director	12/14/07 Date		

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STL STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHOD 8260B AND 8260A

(SUPERSEDES: REVISION 2.4, DATED 09/27/04)

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Approved by:	Dorothy J. Leeson Quality Assurance Manager	6/15/07 Date
Approved by:	William J. Geibel Environmental Health and Safety Coordinator	6-13-07 Date
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1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2. This SOP is applicable to method 8260B. It may also be used for analysis following method 8260A. The associated LIMS method codes are QK (8260B) and MZ (8260A). Ohio VAP projects are distinguished by Program Code 2J. The following Prep Codes are used: 15 (5 mL purge), 25 (low level 5mL purge), 4B (5035, Methanol preservation, EnCore™), 4D (5035, Sodium Bisulfate preservation, EnCore™), 4P (Frozen, EnCore™), M8 (5035A, Frozen Encore™), and 73 (5030A Methanol Prep).

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 40 µg/L for low-level waters, 5 to 200 µg/kg for low-level soils, and 250 to 10,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1 and 3.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2.0 SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Soils are preserved by extracting the volatile analytes into methanol. Soil samples may be preserved with sodium bisulfate or by freezing and purged directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components

which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3.0 DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Refer to the STL QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

3.2.1. A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

3.3.1. Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

3.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in

environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.5.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

3.6.1. CCCs are a representative group of compounds which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

3.7.1. SPCCs are compounds which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the

sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, the sample is diluted.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Cut resistant gloves MUST be worn when opening VOA vials and when doing any other task that presents a strong possibility of getting cut.

5.3. Primary Materials Used

5.3.1. 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
Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure may cause burn if not flushed with water. May cause mild irritation to skin. Prolonged exposure may cause burn if not flushed with water.

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

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with a sticker that reads "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S co-ordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to the EH&S coordinator and the laboratory group leader.

5.7. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the STL Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

5.8. Specific Safety Concerns or Requirements

5.8.1. The gas chromatograph and mass spectrometer 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.

5.8.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.8.3. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.8.4. Sodium bisulfate creates Sulfuric Acid when mixed with water.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger, 0.006 inch ID needle.

6.2. Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.4. Glassware:

6.4.1. Vials: 20 mL with screw caps and Teflon liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipettes: Pasteur, 5 ¾ in.

6.7. pH paper: Wide range, pH 0-14.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity, from cylinders or gas generators, may be used as an

alternative to helium for purge gas.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.9.2. Trap: A variety of traps may be used, depending on the target analytes required. One of the traps used is the Vocarb 3000 trap. Other traps such as the OI 10 may be used if the Quality Control criteria are met. Refer also to instrument operating manuals located within the laboratory.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 20m x 0.18 ID DB-624 with 1 μm film thickness.

6.10.2.2. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS interface: In general direct introduction to the mass spectrometer is used but any interface that achieves all acceptance criteria may be used.

6.10.4. Data System: A computer system that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.1.3. Hydrochloric Acid – (1:1 v/v): Reagent grade or equivalent.

7.1.4. Sodium bisulfate: Reagent grade or equivalent.

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by

more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5 g or 25 g sampler can be used,

depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore™ samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore™ samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore™ sampler per field sample position.

8.5.2. An additional 2 oz plastic bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25 g sample, 5 µL for a nominal 5 g sample.) Refer to Section 17.5 for Michigan project criteria.

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.4 for Michigan project criteria.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.4 for Michigan project criteria.

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional 2 oz plastic bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25 g sample, 5 µL for a nominal 5 g sample.) Refer to Section 17.5 for Michigan project criteria.

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.5 for Michigan project criteria.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.5 for Michigan project criteria.

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (field methanol preserved or using the EnCore™ sampler) procedure, in case the concentration of analytes in the soil is above the calibration range of the low level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.1g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

Note: Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours. The sample must be analyzed within 14 days after sampling and stored at a 45 degree angle in the freezer.

8.7.8. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional 2 oz plastic bottle with no preservative for percent moisture determination, and an additional VOA vial preserved with methanol for the medium level procedure. Depending on the type of soil it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved soils

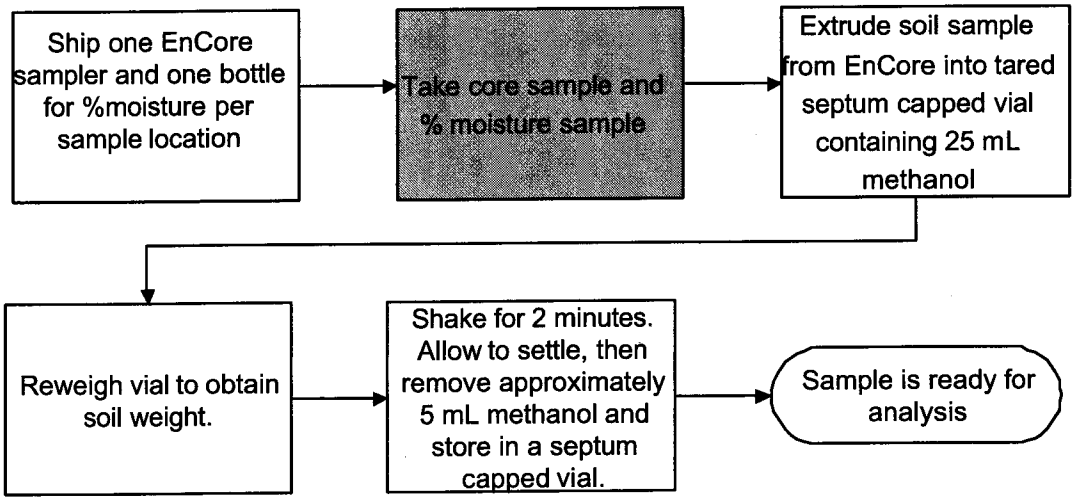
8.8.1. At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab. This is the old procedure based on method 5030A and method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.9. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

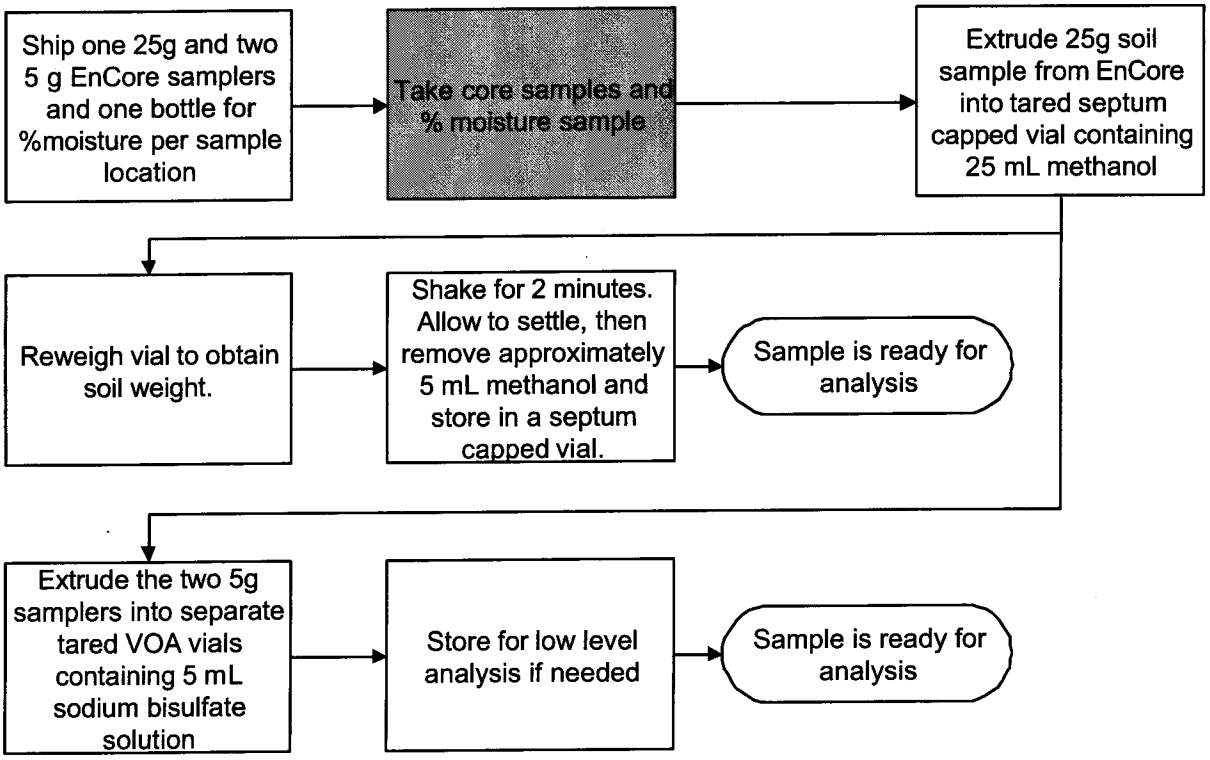
8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.11. A holding blank is stored with the samples. This is analyzed weekly. It is replaced every seven days.

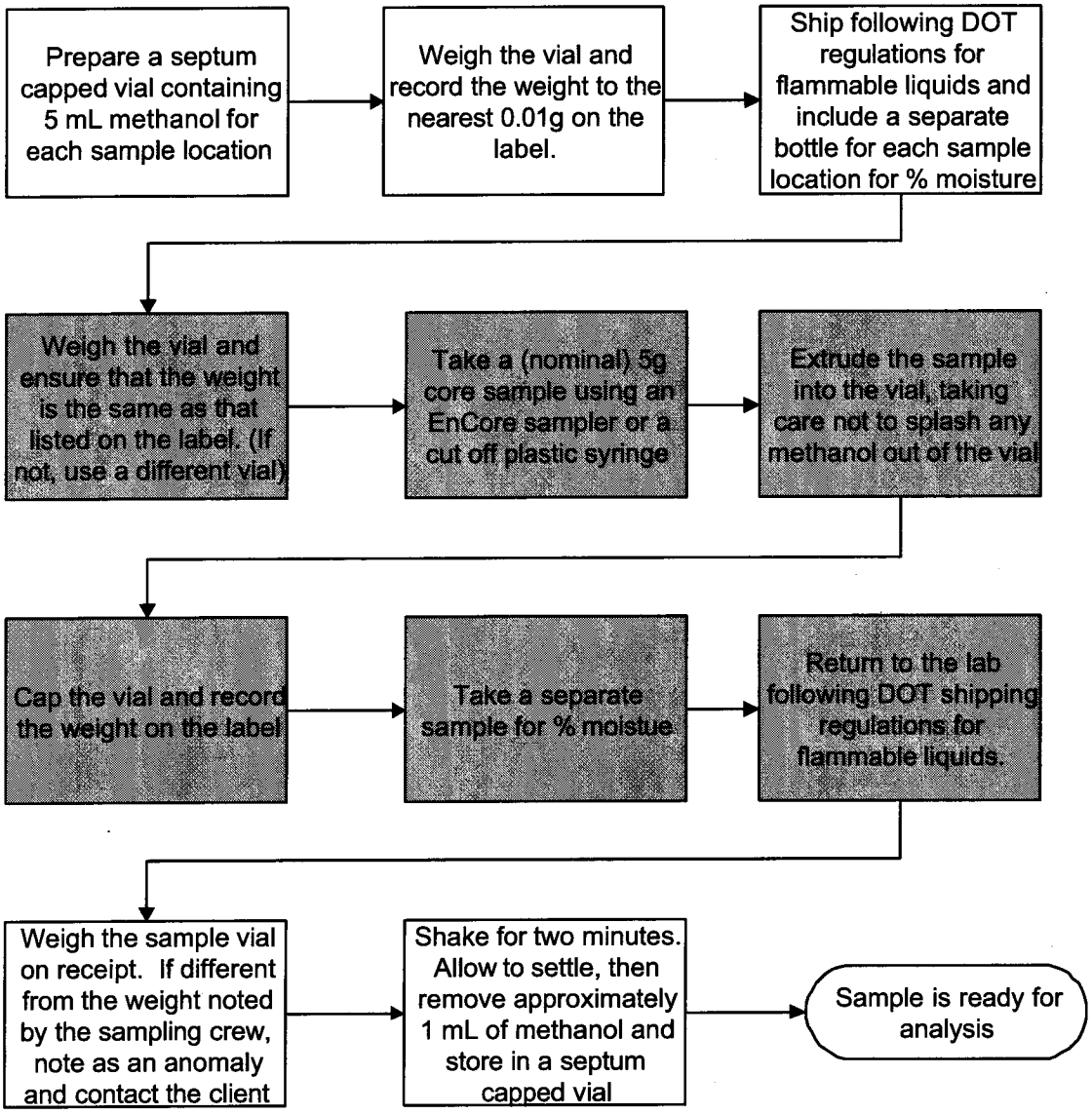
EnCore procedure when low level is not required (field steps in gray)



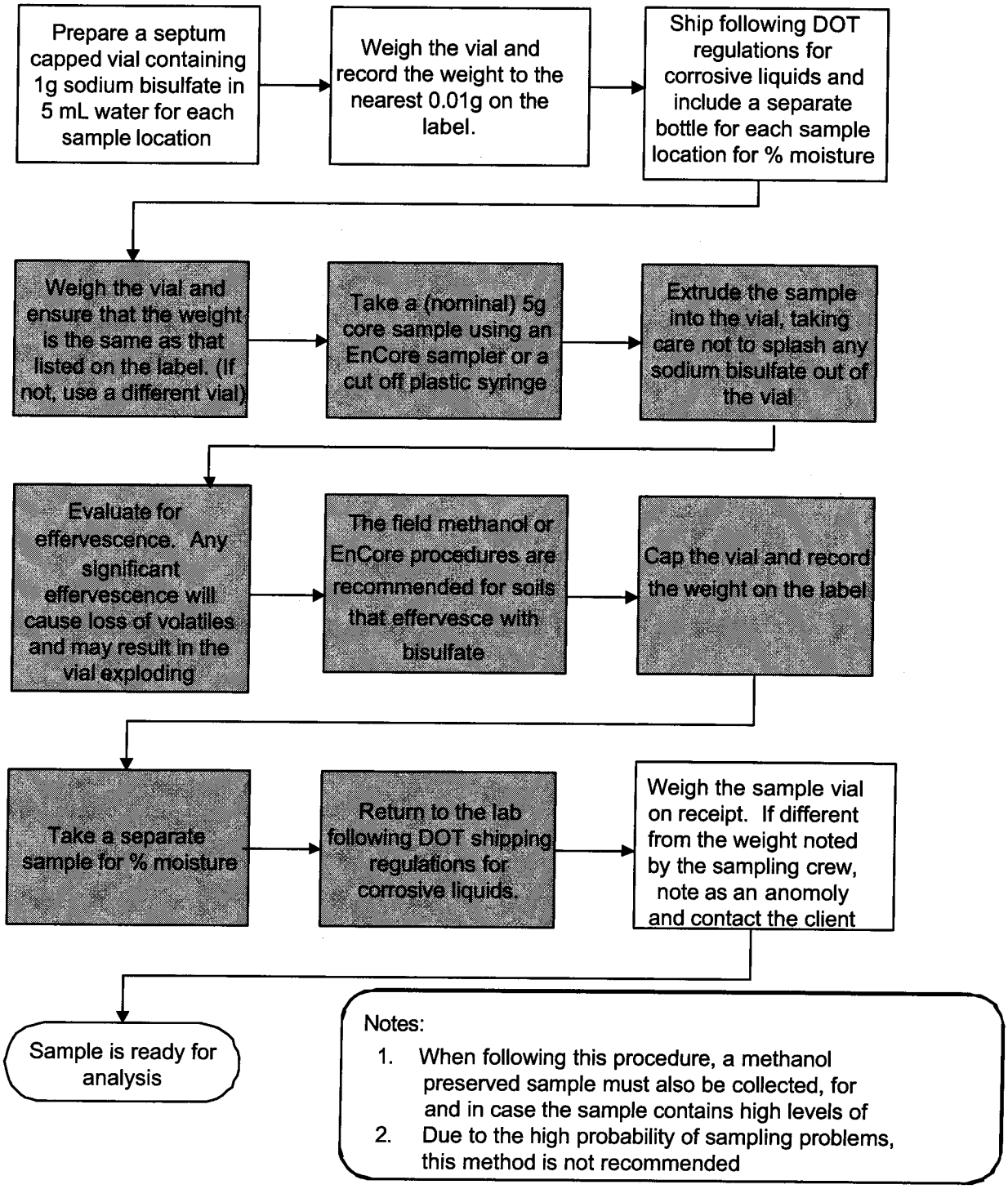
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Control Limits

9.1.1. Control limits are established by the laboratory as described in SOP NC-QA-0018.

9.1.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs (QC Browser program).

9.2. Surrogates

9.2.1. Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 8. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample if there is sufficient volume. If there is insufficient volume, the surrogate is narrated.

It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.2.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.2.3. Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.3. Method Blanks

9.3.1. For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method

blank consists of reagent water. For medium-level volatiles, the method blank consists of the same volume of methanol that was used to prepare the samples. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below). The method blank is acceptable if any compound detected in the blank is present in the associated samples at 10 times the blank level.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9.3.2. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.3.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.3.4. Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.4. Laboratory Control Samples (LCS)

9.4.1. For each batch of samples, analyze a LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.4.2. Refer to the STL QC Program document (QA-003) for further details of the corrective action.

9.4.3. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client. Refer to Section 17.2 for Ohio VAP specific analytes.

9.4.4. If full analyte spike lists are used at the client request, it is possible some compounds in the LCS may interfere with each other. In that case, the lab will quantitate those compounds in the LCS with a secondary ion which is free from interferences.

9.5. Matrix Spikes

9.5.1. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits. See Section 17.2 for Ohio VAP specific analytes.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.6. Non-Conformance and Corrective Action

9.6.1. Any deviations from QC procedures must be documented as a non-conformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system.

10.1.2. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 2 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2. Gas chromatograph suggested temperature program

10.2.1. BFB Analysis

Isothermal:	170°C

10.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C
Final Hold Time:	2.6 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of at least five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a low level 5mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 2 and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained on the Agilent 5973 instruments to use a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6).

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 7. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds

(SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. For any analyte with %RSD >15%, linear or quadratic curve fits may be used. The analyst should consider instrument maintenance to improve the linearity of response. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL , as long as a minimum of 5 points are maintained (6 points for quadratic) and the quantitation range is adjusted accordingly. Otherwise the coefficient of determination, r^2 must be $\geq$ 0.990.

10.4.6.1. Refer to Section 17.2 for specific Ohio VAP criteria.

10.4.7. Weighting of data points

10.4.7.1. In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. The Y-intercept is evaluated to determine calibration acceptability.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. The calibration standards for the initial 5-point calibration for low level soils that are not preserved in sodium bisulfate (i.e. are preserved by freezing, or not preserved) must be heated to 40°C for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40°C. A separate five point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard for analysis of sodium bisulfate preserved samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1 g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary.

Note: This procedure may not be used for Ohio VAP samples.

10.4.11. Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. The recovery for CCC compounds must be ≤ 20%. The recovery for non-CCC compounds must be ≤ 50% with an allowance of up to six compounds > 50%.

10.5. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. A midpoint calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift of all analytes must be ≤ 50% with allowance for up to six target analytes to have % drift > 50%.

10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.3.2. Refer to Table 12 for specific Ohio VAP analytes.

10.5.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.5.3 and 10.5.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Non-Conformance Memo and approved by a Supervisor or Group Leader and QA Manager. If contractually required, the client shall be notified. The Non-Conformance Memo shall be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample then serial dilutions must be made in volumetric flasks.

11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.

11.3.2.2. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.4. Water Samples

11.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.4.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is ≥ 5 µL. Check and document the pH of the remaining sample.

11.4.3. Add 250 ng of each internal and surrogate standard (5 µL of a 50 µg/mL solution, refer to Tables 7and 8). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a 5 mL sample, and a 10 µg/L solution for a 25 mL sample). Inject the sample into the purging chamber.

11.4.3.1. For TCLP samples use 1 mL of TCLP leachate with 4 mL reagent water. (Note that TCLP reporting limits will be 5 times higher than the corresponding aqueous limits).

11.4.4. Purge the sample for eleven minutes (the trap must be below 35°C).

11.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.5. Methanol Extract Soils

11.5.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 1µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1µL will be added to the water in the syringe. Refer to Section 17.5 for Michigan project requirements.

11.6. Liquid wastes that are soluble in methanol and insoluble in water.

11.6.1. Pipet 1 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.6.2. Quickly add 4 mL of methanol, then add 5µL of a 2500 µg/mL surrogate spiking solution to bring the final volume to 5 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 4.9 mL of methanol, 5µL of a 2500 µg/mL surrogate spiking solution, and 0.1 mL of matrix spike solution is used.

11.6.3. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1µL will be added to the water in the syringe.

11.7. Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.7.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.7.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.7.3. Soil samples, which are preserved with sodium bisulfate, must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1 g in 5 mL).

11.7.4. Soil samples, which are preserved by freezing, must be allowed to thaw completely before sample analysis begins.

11.7.5. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.8. Low-Level Solids Analysis using discrete autosamplers, Method 8260A, 5030A.

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5 g. If the sample is contaminated with analytes such that a purge amount less than 0.5 g is appropriate, use the medium level method. For the medium level method, add 5g soil to 5 mL methanol containing the surrogates, mix for two minutes, allow to settle then remove a portion of the methanol and store in a clean Teflon capped vial at 4°C until analysis. Analyze as described in section 11.5.

11.8.3. Connect the purge vessel to the purge and trap device.

11.8.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from 11.5.1.

11.8.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.8.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.8.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.

11.9. Medium-Level Soil/Sediment and Waste Samples

11.9.1. Sediments/soils and waste that are insoluble in methanol.

11.9.1.1. Sediments/soils and waste that are insoluble in methanol.

11.9.1.1.1. Gently mix the contents of the sample container with a narrow metal or wood spatula. Weigh 5 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.9.1.1.2. Quickly add 5 mL of methanol, and 5µL of 2500 µg/mL

surrogate spiking solution to bring the final volume of methanol to 5 mL. For an LCS or MS/MSD sample add 4.9 mL of methanol, 5µL of surrogate spike solution, and 0.1 mL of matrix spike solution. Cap the vial and shake or vortex to mix thoroughly.

Note: Sections 11.9.1.1.1 and 11.9.1.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.10. Initial review and corrective actions

11.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.10.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is reanalyzed to confirm. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected.

11.10.3. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once, at the same dilution as the original run, to demonstrate matrix effect, but reanalysis at a dilution could be considered for severe matrix effect.

11.11. Dilutions

11.11.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.11.2 Guidance for Dilutions Due to Matrix

11.11.2.1 If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.11.3 Reporting Dilutions

11.11.3.1 The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of

the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations

12.3.1. Response factor (RF)

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N-1}}$$

Where:

X_i = Value of X at i through N

N = Number of points

X = Average value of X_I

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\textbf{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ **= Mean of RF values in the curve**

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\% \text{ Drift} = \frac{C_{expecte} - C_{found}}{C_{expecte}} \times 100$$

Where:

$C_{expecte}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

12.3.5.1 Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.5.2 Calculation of concentration using Average Response Factors:

Equation 5

$$\textbf{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.3 Calculation of concentration using Linear fit:

Equation 6

$$\textbf{Concentration } \mu g / L = A + Bx$$

12.3.5.3. Calculation of concentration using Quadratic fit:

Equation 7

$$\textbf{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10
A is a constant defined by the intercept
B is the slope of the curve
C is the curvature

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

X = ug/L

$\mathbf{A_x}$ = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

$\mathbf{A_{is}}$ = Area of the characteristic ion for the internal standard

$\mathbf{I_s}$ = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

$\mathbf{V_o}$ = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

X = ug/kg

$\mathbf{A_x}$, $\mathbf{I_s}$, $\mathbf{D_f}$, $\mathbf{A_{is}}$, same as for water.

$\mathbf{V_t}$ = Volume of total extract, mL (Typically 25 mL)

$\mathbf{V_a}$ = Volume of extract added for purging, µL

$\mathbf{W_s}$ = Weight of sample extracted, g

$$\mathbf{D} = \frac{100 - \%\text{moisture}}{100}$$

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

X = ug/kg

$\mathbf{A_x}$, $\mathbf{I_s}$, $\mathbf{A_{is}}$, same as for water.

D is as for medium level soils

$\mathbf{W_s}$ = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

$\mathbf{A_x}$ = Area in the total ion chromatogram for the compound being measured

$\mathbf{A_{is}}$ = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result
SR = Sample result
SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD:

Equation 12

$$\mathbf{RPD} = \frac{|\mathbf{MSR - MSDR}|}{\frac{1}{2}(\mathbf{MSR + MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: S-Q-003 and NC-QA-0021. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method. The non-standard compound must be detected in the reporting limit standard to be acceptable.

13.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

13.2. Initial Demonstration

13.2.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.3.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.2. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

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

15.3.1. **Acidic material from the auto-sampler**: Waste stream must be collected and neutralized before discharge to a sewer system if the pH is less than 4.

15.3.2. **Methanol waste from rinses and standards:** Methanol waste is discarded as a

flammable liquid in a solvent waste container identified as "Flammable Liquid Waste."

15.3.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The waste coordinator handles crushing the vials and proper disposal.

15.3.4. **Solid samples** - Stirbars are removed from the sample. The contents of the vial are poured into a beaker and the soil allowed to settle out. The soil is disposed of in the solid waste container.

16. REFERENCES

16.1. References

16.1.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.1.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994.

16.1.3. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030B, Rev 2, December 1996.

16.1.4. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996.

16.1.5. Corporate Quality Management Plan (QMP), current version.

16.1.6. STL Laboratory Quality Manual (LQM), current version.

16.1.7. STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, S-Q-003 and NC-QA-0021

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.6. Standards and Reagents, NC-QA-0017

16.2.7. Laboratory Holding Blanks, NC-QA-0020

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2. The following are protocols that must be followed when analyzing OhioVAP samples:

- Sections 9.4 and 9.5: n-Hexane must be spiked and reported for both the LCS and MS/MSD.
- Sections 10.4.6: All analytes must have a %RSDs $\leq$ 15%. Corrective action must be completed for any compounds failing the <15% requirement.
- Section 11.1 is not to be performed.
- Section 11.10.2: For OhioVAP projects, the laboratory will reanalyze any sample where the internal standard fails and there is no evidence of matrix interference.

17.3. The following are protocols that must be followed when analyzing BP Oil – Lima Refinery RFI work plan.

- Section 8.1 STL will continue to follow the 14 day holding time specified in the Corporate SOP.
- Delete for this project Section 8.3 At specific client request, unpreserved soil samples may be accepted.
- Delete for this project Section 8.8.1 At specific client request unpreserved soils packed into

glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A. It is no longer included and is likely to generate results that are biased low, possibly by more than an order of magnitude.

- Modify Section 8.5 For the purpose of this project, the soil/methanol mixture may be stored for two days prior to analysis.
- Modify Section 8. For the purpose of this project, the soil/methanol mixture may be stored for two days prior to analysis.
- Modify (per discussion with Region V representative) to Section 10.4.6 Compounds with %RSD >15% are to be calibrated using an alternate calibration technique (e.g. linear or quadratic calibration curve). For poor responders, the alternate calibration technique requirements may not be met either. This sentence is added for those cases.If the correlation coefficient is < 0.990, then any hit for these compounds must be flagged as estimated.
- Modify Section 10.4.2 It is necessary to analyze the Appendix IX standard separately from the primary standard due to the presence of xylene solvent in the Appendix IX standard. Alternatively, STL will purchase the Appendix IX standard in a solvent other than xylene.
- Modify Section 10.4.9 For this project, this section will be modified to comply with the requirement of adding methanol to the calibration standards so that those standards contain the same amount of methanol as the diluted soil extracts.
- Modify Table 6
- For the project specific SOP, acetonitrile will be removed from table 6, page 49 and appended onto table 5, page 48. Acetonitrile will be calibrated as part of the STL primary standard, using a separate acetonitrile standard. This will ensure that the calibration curve for acetonitrile will be done free from any interference from allyl chloride.

17.4. The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.

17.4.1. Modify Section 8.5.4 and 8.6.8 (add 25 uL of 2500 ug/mL surrogate solution for a nominal 25 g sample).

17.4.2. Modify Section 8.5.5 and 8.6.9 (add 500 uL of 50 ug/mL spike solution for a nominal 25 g sample).

17.4.3. Modify Section 8.5.6 and 8.6.10 (add 100 uL of 50 ug/mL spike solution for a nominal 25g sample).

17.4.4. Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended

calibration levels in water of 0.5 ug/L, 1 ug/L, 5 ug/L, 10 ug/L, 20 ug/L, and 50 ug/L. Some analytes are prepared at higher concentrations.

17.4.5. Samples for Michigan projects frequently require calibration for 2-Methylnaphthalene. Recommended calibration levels for this compounds are 2 ug/L, 10 ug/L, 20 ug/L, 40 ug/L and 80 ug/L.

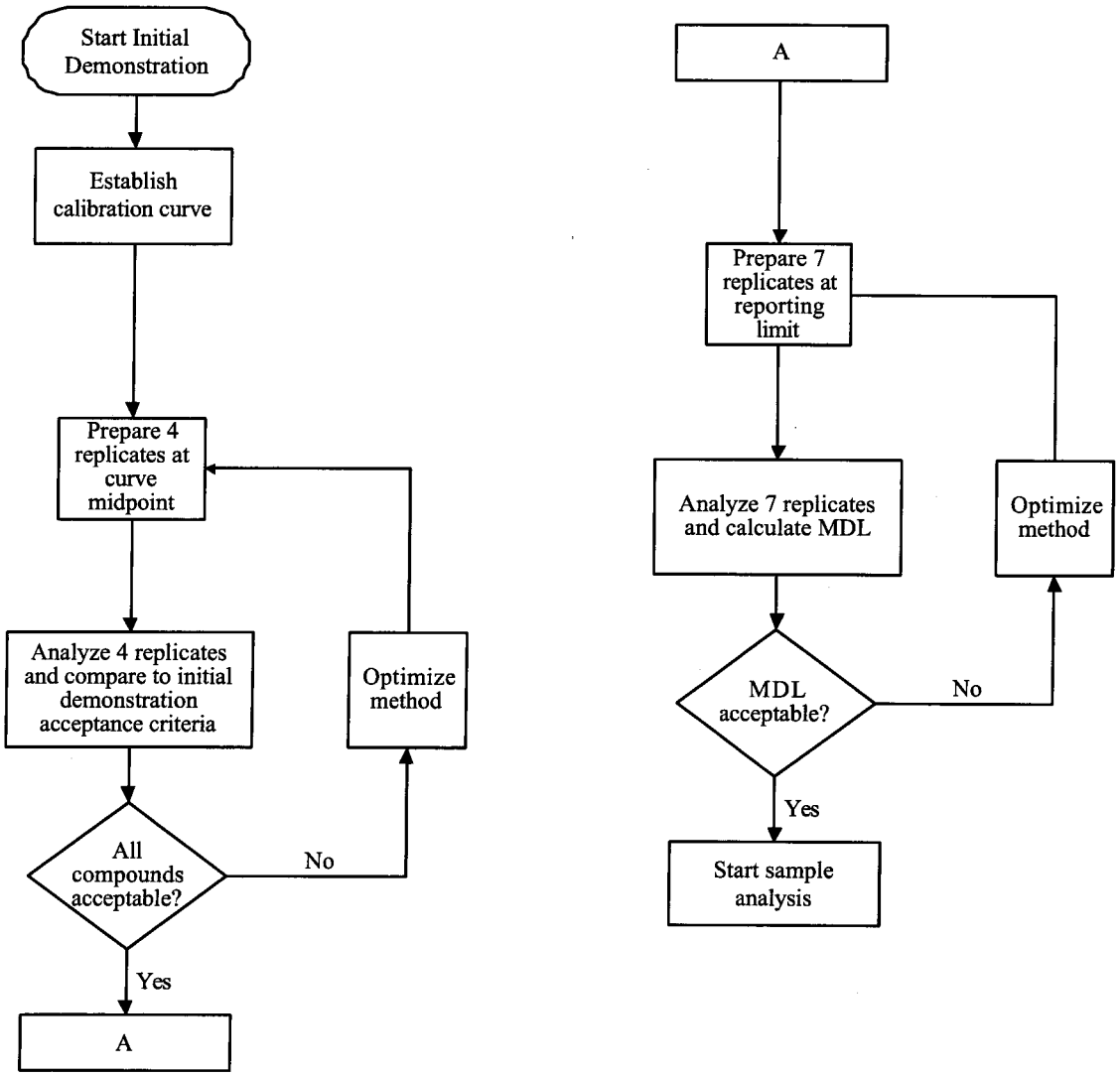


Table 1 - STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dichlorodifluoromethane	75-71-8	5	1	5	250	1200
Chloromethane	74-87-3	5	1	5	250	1200
Bromomethane	74-83-9	5	1	5	250	1200
Vinyl chloride	75-01-4	5	1	5	250	1200
Chloroethane	75-00-3	5	1	5	250	1200
Trichlorofluoromethane	75-69-4	5	1	5	250	1200
Acrolein	107-02-8	100	20	100	5000	12000
Acetone	67-64-1	20	10	20	1000	2500
Trichlorotrifluoroethane	76-13-1	5	1	5	250	620
Iodomethane	74-88-4	5	1	5	250	620
Carbon disulfide	75-15-0	5	1	5	250	620
Methylene chloride	75-09-2	5	1	5	250	620
tert-Butyl alcohol	75-65-0	200	50	200	10,000	25000
1,1-Dichloroethene	75-35-4	5	1	5	250	620
1,1-Dichloroethane	75-34-3	5	1	5	250	620
trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250	310
Acrylonitrile	107-13-1	100	20	100	5000	12000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	1000	2500
Hexane	110-54-3	5	1	5	250	620
cis-1,2-Dichloroethene	156-59-2	5	1	5	250	620
1,2-Dichloroethene (Total)	540-59-0	10	2	10	500	620
Tetrahydrofuran	109-99-9	20	5	20	1000	2500
Chloroform	67-66-3	5	1	5	250	620
1,2-Dichloroethane	107-06-2	5	1	5	250	620
Dibromomethane	74-95-3	5	1	5	250	620
2-Butanone	78-93-3	20	5	20	1000	2500
1,4-Dioxane	123-91-1	500	200	500	25000	62000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	620
Carbon tetrachloride	56-23-5	5	1	5	250	620
Bromodichloromethane	75-27-4	5	1	5	250	620
1,2-Dichloropropane	78-87-5	5	1	5	250	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	620
Trichloroethene	79-01-6	5	1	5	250	620

Table 1 - STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dibromochloromethane	124-48-1	5	1	5	250	620
1,2-Dibromoethane	106-93-4	5	1	5	250	620
1,2,3-Trichloropropane	96-18-4	5	1	5	250	620
1,1,2-Trichloroethane	79-00-5	5	1	5	250	620
Benzene	71-43-2	5	1	5	250	620
Ethylmethacrylate	97-63-2	5	1	5	250	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	620
Bromoform	75-25-2	5	1	5	250	620
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	2500
2-Hexanone	591-78-6	20	5	20	1000	2500
Tetrachloroethene	127-18-4	5	1	5	250	620
Toluene	108-88-3	5	1	5	250	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250	620
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000	6200
Vinyl acetate	108-05-4	10	2	10	500	1200
Chlorobenzene	108-90-7	5	1	5	250	620
Ethylbenzene	100-41-4	5	1	5	250	620
Styrene	100-42-5	5	1	5	250	620
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	620
m and p Xylenes		10	2	10	500	1200
o-xylene	95-47-6	5.0	1	5	250	620
Total xylenes	1330-20-7	10	2	10	500	1200
1,3-Dichlorobenzene	541-73-1	5	1	5	250	620
1,4-Dichlorobenzene	106-46-7	5	1	5	250	620
1,2-Dichlorobenzene	95-50-1	5	1	5	250	620
2,2-Dichloropropane	590-20-7	5	1	5	250	
Bromochloromethane	74-97-5	5	1	5	250	
1,1-Dichloropropene	563-58-6	5	1	5	250	
Bromodichloromethane	75-27-4	5	1	5	250	
1,2-Dichloropropane	78-87-5	5	1	5	250	
1,3-Dichloropropane	142-28-9	5	1	5	250	
Isopropylbenzene	98-82-8	5	1	5	250	
Bromobenzene	108-86-1	5	1	5	250	
n-Propylbenzene	103-65-1	5	1	5	250	
2-Chlorotoluene	95-49-8	5	1	5	250	

Table 1 - STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
4-Chlorotoluene	106-43-4	5	1	5	250	
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	
Tert-Butylbenzene	98-06-6	5	1	5	250	
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250	
Sec-butylbenzene	135-98-8	5	1	5	250	
4-Isopropyltoluene	99-87-6	5	1	5	250	
n-Butylbenzene	104-51-8	5	1	5	250	
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250	
Napthalene	91-20-3	5	1	5	250	
Hexachlorobutadiene	87-68-3	5	1	5	250	
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250	
Acetonitrile	75-05-8	100	20	100	5000	
Cyclohexane	110-82-7	10	1	10	500	
Methyl Acetate	79-20-9	10	10	10	500	
Methyl cyclohexane	108-87-2	10	1	10	500	

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2 - STL Primary Standard Calibration Levels, 5 mL purge

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	50	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200
Dibromochloromethane	5	20	50	100	200

Table 2 - STL Primary Standard Calibration Levels, 5 mL purge

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
Acetonitrile	50	200	500	1000	2000
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200
2,2-Dichloropropane	5	20	50	100	200
Bromochloromethane	5	20	50	100	200
1,1-Dichloropropene	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
1,3-Dichloropropane	5	20	50	100	200
Isopropylbenzene	5	20	50	100	200
Bromobenzene	5	20	50	100	200
n-Propylbenzene	5	20	50	100	200
2-Chlorotoluene	5	20	50	100	200
4-Chlorotoluene	5	20	50	100	200
1,3,5-Trimethylbenzene	5	20	50	100	200
tert-Butylbenzene	5	20	50	100	200
1,2,4-Trimethylbenzene	5	20	50	100	200
sec-butylbenzene	5	20	50	100	200
4-Isopropyltoluene	5	20	50	100	200

Table 2 - STL Primary Standard Calibration Levels, 5 mL purge

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
n-Butylbenzene	5	20	50	100	200
1,2,4-Trichlorobenzene	5	20	50	100	200
Napthalene	5	20	50	100	200
Hexachlorobutadiene	5	20	50	100	200
1,2,3-Trichlorobenzene	5	20	50	100	200

Table 2A - STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromofluoromethane (Surrogate)	1	5	10	20	40
1,2-Dichloroethane-d4 (Surrogate)	1	5	10	20	40
Toluene-d8 (Surrogate)	1	5	10	20	40
Bromofluorobenzene (Surrogate)	1	5	10	20	40
Dichlorodifluoromethane	1	5	10	20	40
Chloromethane	1	5	10	20	40
Vinyl Chloride	1	5	10	20	40
Bromomethane	1	5	10	20	40
Chloroethane	1	5	10	20	40
Trichlorofluoromethane	1	5	10	20	40
Acrolein	10	50	100	200	400
Acetone	2	10	20	40	80
1,1-Dichloroethene	1	5	10	20	40
Trichlorotrifluoroethane	1	5	10	20	40
Iodomethane	1	5	10	20	40
Carbon Disulfide	1	5	10	20	40
Methylene Chloride	1	5	10	20	40
Acetonitrile	10	50	100	200	400
Acrylonitrile	10	50	100	200	400
Methyl tert-butyl ether	1	5	10	20	40
trans-1,2-Dichloroethene	1	5	10	20	40
Hexane	1	5	10	20	40
Vinyl acetate	1	5	10	20	40
1,1-Dichloroethane	1	5	10	20	40
tert-Butyl Alcohol	20	100	200	400	800
2-Butanone	2	10	20	40	80
cis-1,2-dichloroethene	1	5	10	20	40
2,2-Dichloropropane	1	5	10	20	40
Bromochloromethane	1	5	10	20	40
Chloroform	1	5	10	20	40
Tetrahydrofuran	1	5	10	20	40
1,1,1-Trichloroethane	1	5	10	20	40
1,1-Dichloropropene	1	5	10	20	40
Carbon Tetrachloride	1	5	10	20	40
1,2-Dichloroethane	1	5	10	20	40
Benzene	1	5	10	20	40
Trichloroethene	1	5	10	20	40
1,2-Dichloropropane	1	5	10	20	40
1,4-Dioxane	50	250	500	1000	2000

Table 2A - STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromomethane	1	5	10	20	40
Bromodichloromethane	1	5	10	20	40
2-Chloroethyl vinyl ether	2	10	20	40	80
cis-1,3-Dichloropropene	1	5	10	20	40
4-Methyl-2-pentanone	2	10	20	40	80
Toluene	1	5	10	20	40
trans-1,3-Dichloropropene	1	5	10	20	40
Ethyl Methacrylate	1	5	10	20	40
1,1,2-Trichloroethane	1	5	10	20	40
1,3-Dichloropropane	1	5	10	20	40
Tetrachloroethene	1	5	10	20	40
2-Hexanone	2	10	20	40	80
Dibromochloromethane	1	5	10	20	40
1,2-Dibromoethane	1	5	10	20	40
Chlorobenzene	1	5	10	20	40
1,1,1,2-Tetrachloroethane	1	5	10	20	40
Ethylbenzene	1	5	10	20	40
m + p-Xylene	2	10	20	40	80
Xylene-o	1	5	10	20	40
Styrene	1	5	10	20	40
Bromoform	1	5	10	20	40
Isopropylbenzene	1	5	10	20	40
1,1,2,2-Tetrachloroethane	1	5	10	20	40
1,4-Dichloro-2-butene	1	5	10	20	40
1,2,3-Trichloropropane	1	5	10	20	40
Bromobenzene	1	5	10	20	40
n-Propylbenzene	1	5	10	20	40
2-Chlorotoluene	1	5	10	20	40
1,3,5-Trimethylbenzene	1	5	10	20	40
4-Chlorotoluene	1	5	10	20	40
tert-Butylbenzene	1	5	10	20	40
1,2,4-Trimethylbenzene	1	5	10	20	40
sec-Butylbenzene	1	5	10	20	40
4-Isopropyltoluene	1	5	10	20	40
1,3-Dichlorobenzene	1	5	10	20	40
1,4-Dichlorobenzene	1	5	10	20	40
n-Butylbenzene	1	5	10	20	40
1,2-Dichlorobenzene	1	5	10	20	40
1,2-Dibromo-3-chloropropane	1	5	10	20	40
1,2,4-Trichlorobenzene	1	5	10	20	40

Table 2A - STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Hexachlorobutadiene	1	5	10	20	40
Naphthalene	1	5	10	20	40
1,2,3-Trichlorobenzene	1	5	10	20	40
Cyclohexane	1	5	10	20	40
Methyl Acetate	2	10	20	40	80
Methylcyclohexane	1	5	10	20	40
1,3,5-Trichlorobenzene	1	5	10	20	40

[1] 25 mL purge samples analyzed at 5 mL purge on more sensitive equipment.

Table 3 - STL Appendix IX Standard and Reporting Limits, 5 mL purge

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	Low Level 5mL purge water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Dichlorofluoromethane	75-43-4	10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	2	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	2	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	4	10	500
Cyclohexanone	108-94-1	50	20	50	2500
Isopropylbenzene	98-82-8	5	1	5	250
2-Methylnaphthalene (Michigan only)	91-57-6	NA	5	NA	330

Table 4

Recommended/STL Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
2-Methylnaphthalene (Michigan only)	2	10	20	40	80

Table 5 - Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
Dichlorodifluoromethane	75-71-8			X	X
Chloromethane	74-87-3	X		X	X
Bromomethane	74-83-9	X		X	X
Vinyl chloride	75-01-4	X	X	X	X
Chloroethane	75-00-3	X		X	X
Trichlorofluoromethane	75-69-4			X	X
Acrolein	107-02-8				X
Acetone	67-64-1	X		X	X
Trichlorotrifluoroethane	76-13-1				
Iodomethane	74-88-4				X
Carbon disulfide	75-15-0	X		X	X
Methylene chloride	75-09-2	X		X	X
tert-Butyl alcohol	75-65-0				
1,1-Dichloroethene	75-35-4	X	X	X	X
1,1-Dichloroethane	.75-34-3	X		X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X
Total 1,2-Dichloroethene		X		X	X
Acrylonitrile	107-13-1				X
Methyl *tert*-butyl ether (MTBE)	1634-04-4			X	
Hexane	110-54-3				
cis-1,2-Dichloroethene	156-59-2	X		X	
Tetrahydrofuran	109-99-9				
Chloroform	67-66-3	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X
Dibromomethane	74-95-3				X
2-Butanone	78-93-3	X	X	X	X
1,4-Dioxane	123-91-1				X
1,1,1-Trichloroethane	71-55-6	X		X	X
Carbon tetrachloride	56-23-5	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X
1,2-Dichloropropane	78-87-5	X		X	X
cis-1,3-Dichloropropene	10061-01-5	X		X	X
Trichloroethene	79-01-6	X	X	X	X
Dibromochloromethane	124-48-1	X		X	X
1,2-Dibromoethane	106-93-4			X	X
1,2,3-Trichloropropane	96-18-4				X
1,1,2-Trichloroethane	79-00-5	X		X	X
Benzene	71-43-2	X	X	X	X
Ethylmethacrylate	97-63-2				X
trans-1,3-Dichloropropene	10061-02-6	X		X	X

Table 5 - Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
Bromoform	75-25-2	X		X	X
4-Methyl-2-pentanone	108-10-1	X		X	X
2-Hexanone	591-78-6	X		X	X
Tetrachloroethene	127-18-4	X	X	X	X
Toluene	108-88-3	X		X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X
2-Chloroethyl vinyl ether	110-75-8				
Vinyl acetate	108-05-4				X
Chlorobenzene	108-90-7	X	X	X	X
Ethylbenzene	100-41-4	X		X	X
Styrene	100-42-5	X		X	X
t-1,4-Dichloro-2-butene	110-57-6				X
m and p Xylenes		X		X	X
o-xylene	95-47-6	X		X	X
Total xylenes	1330-20-7	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	
1,4-Dichlorobenzene	106-46-7	X		X	
1,2-Dichlorobenzene	95-50-1	X		X	
Cyclohexane	110-82-7	X		X	
Methyl Acetate	79-20-9	X		X	
Methyl cyclohexane	108-87-2	X		X	
Isopropylbenzene	98-82-8			X	
1,2-Dibromo-3-chloropropane	96-12-8			X	X
1,2,4-Trichlorobenzene	120-82-1			X	
Acetonitrile	75-05-8				X
1,1,1,2-Tetrachloroethane	630-20-6				X
2,2-Dichloropropene	594-20-7				
Bromochloromethane	74-97-5				
1,1-Dichloropropene	563-58-6				
1,3-Dichloropropane	142-28-9				
Bromobenzene	108-86-1				
n-Propylbenzene	103-65-1				
2-Chlorotoluene	95-49-8				
1,3,5-Trimethylbenzene	108-67-8				
4-Chlorotoluene	106-43-4				
tert-Butylbenzene	98-06-6				
1,2,4-Trimethylbenzene	95-63-6				
sec-Butylbenzene	135-98-8				
4-Isopropyltoluene	99-87-6				
n-Butylbenzene	104-51-8				

Table 5 - Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
Hexachlorobutadiene	87-68-3				
Naphthalene	91-20-3				
1,2,3-Trichlorobenzene	87-61-6				
1,3,5-Trichlorobenzene	108-70-3				

Table 6

Reportable Analytes for STL Standard Tests, Appendix IX standard

Compound	Number	Appendix IX
Allyl Chloride	107-05-1	X
Dichlorofluoromethane	75-43-4	
Isopropyl ether	108-20-3	
Chloroprene	126-99-8	X
n-Butanol	71-36-3	
Propionitrile	107-12-0	X
Methacrylonitrile	126-98-7	X
Isobutanol	78-83-1	X
Methyl methacrylate	80-62-6	X
Ethyl ether	60-29-7	
Ethyl Acetate	141-78-6	
2-Nitropropane	79-46-9	
Cyclohexanone	108-94-1	

Table 7 - Internal Standards

Compound	Standard Concentration µg/mL	Quantitation ion (5 mL purge)
Fluorobenzene	50	96
Chlorobenzene-d5	50	117
1,4-Dichlorobenzene-d4	50	152

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8 - Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	50
Dibromofluoromethane	50
Toluene-d_8	50
4-Bromofluorobenzene	50

Notes:

1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

2) Recovery limits for surrogates are generated from historical data and are maintained by the QA Dept.

Table 9 - Matrix Spike / LCS Control Compounds

Compound	Standard Concentration µg/mL
1,1,1-Trichloroethane	50
1,1,2,2-Tetrachloroethane	50
1,1,2-Trichloro-1,2,2-trifluoroethane	50
1,1,2-Trichloroethane	50
1,1-Dichloroethane	50
1,1-Dichloroethene	**50**
1,1-Dichloropropene	50
1,2,3-Trichlorobenzene	50
1,2,3-Trichloropropane	50
1,2,4-Trichlorobenzene	50
1,2,4-Trimethylbenzene	50
1,2-Dibromo-3-chloropropane	50
1,2-Dibromoethane	50
1,2-Dichlorobenzene	50
1,2-Dichloroethane	50
1,2-Dichloroethene (total)	100
1,2-Dichloropropane	50
1,3,5-Trimethylbenzene	50
1,3-Dichlorobenzene	50
1,3-Dichloropropane	50
1,4-Dichlorobenzene	50
2,2-Dichloropropane	50
2-Butanone	50
2-Chloroethyl Vinyl Ether	100
2-Chlorotoluene	50
2-Hexanone	50
4-Chlorotoluene	50
4-Methyl-2-pentanone	50
Acetone	50
Acetonitrile	500
Acrolein	500
Acrylonitrile	500
Benzene	**50**
Bromobenzene	50
Bromochloromethane	50
Bromodichloromethane	50
Bromoform	50
Bromomethane	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	**50**

Table 9 - Matrix Spike / LCS Control Compounds

Compound	Standard Concentration µg /mL
Chloroethane	50
Chloroform	50
Chloromethane	50
cis-1,2-Dichloroethene	50
cis-1,3-Dichloropropene	50
Cyclohexane	50
Dibromochloromethane	50
Dibromomethane	50
Dichlorodifluoromethane	50
Ethylbenzene	50
Hexachlorobutadiene	50
Iodomethane	50
Isopropylbenzene	50
Isopropylether	50
Methyl acetate	50
Methyl tert-butyl ether (MTBE)	50
Methylcyclohexane	50
Methylene chloride	50
Naphthalene	50
n-Butylbenzene	50
n-Hexane (Ohio VAP only)	50
n-Propylbenzene	50
p-Isopropyltoluene	50
sec-Butylbenzene	50
Styrene	50
tert-Butylbenzene	50
Tetrachloroethene	50
Toluene	**50**
trans-1,2-Dichloroethene	50
trans-1,2-Dichloroethene	50
trans-1,3-Dichloropropene	50
Trichloroethene	**50**
Trichlorofluoromethane	50
Vinyl Acetate	50
Vinyl chloride	50
Xylenes (total)	150

Notes: 1) 5 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by QA Dept.

3) Analytes in **BOLD** denote control analytes.

Table 10 - BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11 - SPCC Compounds and Minimum Response Factors

Compound	8260B, 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 12 - CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0
n-Hexane (Ohio VAP only)	<30.0	<20.0

Table 13 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	

Table 13 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52

Table 13 - Characteristic Ions

Compound	Primary*	Secondary	Tertiary
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	
Cyclohexane	56	69	84
Methyl Acetate	43	74	
Methyl cyclohexane	83	55	98

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

Title: EXTRACTION OF ORGANIC COMPOUNDS FROM WATERS AND SOILS, BASED ON SW846 3500 SERIES, 3600 SERIES, AND 600 SERIES METHODS

Approvals (Signature/Date):

Technology Specialist	Date 3/3/08	Health & Safety Coordinator	Date 3-6-08
Quality Assurance Manager	Date 3/6/08	Laboratory Director	Date 3/4/08
Technical Director	Date 3/12/08		

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1. SCOPE AND APPLICATION

1.1 This SOP describes procedures for preparation (extraction) of semivolatile organic analytes in aqueous, TCLP leachate, and soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS). The procedures are based on SW-846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1.1. Extraction procedures for the following determinative methods are covered: 8081A, 8082, 8270C, 8015B, 608, and 625

1.1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.1.3. Other extraction procedures (PFE, SPE, etc.) may be used but are not currently covered in this SOP.

1.1.4. The applicable LIMS method codes are: QJ (8081A), QL (8270C), QH (8082), DM (608), DP (625), KI (8015B)

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction

A measured volume of sample, typically 1 liter, is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2. Continuous Liquid/Liquid Extraction

A measured volume of sample, typically 1 liter, is placed into a continuous liquid/liquid extractor, adjusted, if necessary, to a specific pH and extracted with the appropriate solvent for 18-24 hours.

2.3. Sonication Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate until free flowing. This is solvent extracted three times using an ultrasonic horn.

2.4. Soxhlet Extraction (Accelerated and Traditional)

A 30 g sample is mixed with anhydrous sodium sulfate until free flowing. This is extracted with refluxing solvent.

2.5. Concentration

Procedures are presented for drying and concentration of the extract to final volume for analysis.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Lab Specific Addendum to the CSM, and this document.

5.2. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.

5.3. The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

5.4. 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
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 2 Mg/M3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. 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 (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	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. 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.
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of Methylene chloride is spilled,

evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

5.9. 3510 Separatory Funnel

5.9.1. 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, periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, the use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

5.10. 3520 Extraction Continuous Liquid/Liquid

5.10.1. All personnel are to ensure liquid-liquid area is clear of unnecessary items. Heating mantles used with liquid- liquid extractions generate temperatures that could ignite some materials that come in contact with the heating mantles.

5.10.2. Ensure all solvents are away from liquid-liquid extractor. Increased temperatures near solvents can cause the pressure in the containers to increase.

5.10.3. Ensure all boiling flasks have cooled to room temperature before disconnecting liquid-liquid bodies from boiling flasks to prevent any burns.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware should be cleaned per Glassware Washing, SOP NC-QA-0014.

6.2.1. Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Sep Fun.	CLLE	Soni	Sox	Conc
Separatory Funnel: 2 L	√				
Separatory Funnel Rack	√				
Balance: >1400 g capacity, accurate ±1 g	√	√	√	√	
pH indicator paper, Ranges: 0-14, 7.5-14, 0-6, and 3.8-5.5	√	√			
Graduated cylinder: 1 liter. (other sizes may be used as needed)	√	√			√
Erlenmeyer Flask: 125 & 300 mL (other sizes optional)	√	√	√		√
Centrifuge	√				
Methylene Chloride Collection Tank	√	√			
Initial Volume Template	√	√			
Solvent Dispenser Pump or 100 mL Graduated Cylinder	√	√	√	√	√
Continuous Liquid/Liquid Extractor		√			
Round or flat Bottom: 250, 500 mL or 1 L		√		√	
Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent)		√		√	√
Cooling Condensers		√		√	
Heating Mantle: Rheostat controlled		√		√	
Auto-timer for heating mantle		√		√	
Beakers: 250 & 400 mL, graduated	√	√	√		√
450mL wide-mouth glass jars	√		√		√
Balance: >100 g capacity, accurate ±0.1 g	√	√	√	√	√
Soxhlet Extractor				√	
Cellulose and Glass Thimbles				√	
Accelerated Soxhlet Extractor (Soxtherm-trade name)				√	
Sonicator (at least 300 watts)			√		
Sonicator horn, 3/4 inch			√		
Kuderna-Danish (K-D) Apparatus: 500 mL					√
Concentrator Tube: 10 mL, attached to K-D with clips					√
Snyder Column: Three-ball macro					√
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.					√
Vials: Glass, 2 mL, 4 mL, and 10 mL capacity with Teflon®-lined screw-cap					√
Nitrogen Blowdown Apparatus					√
Nitrogen: reagent grade.					√
Culture tubes: 10 mL, 16 mmx100 mm					√
Syringe: 1 mL	√	√	√	√	
Glass Wool	√	√	√	√	
Glass Funnel: 75 X 75 mm	√	√	√	√	√
Disposable Pipets, 5 ¾ in, and 9in.	√	√	√	√	√
Aluminum foil	√	√	√	√	√
Paper Towels	√	√	√	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep Fun.	CLLE	Soni	Sox	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	√	√			
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	√			
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	√	√			
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	√			
Hydrochloric Acid (HCl)			√		
Organic free reagent water.	√	√			
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√	√	√	√
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√	√	√	√
Acetone, Methylene Chloride: Used for cleaning	√	√	√	√	√

7.2. Standards

7.2.1. Stock Standards

Stock standards are purchased as certified solutions. Semivolatile stock standards are stored at $\leq$ 6°C. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampoule is opened if purchased.) Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards.

Spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 Extraction is initiated within seven days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2 For TCLP leachates, extraction is initiated within seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3. Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Insufficient Sample

9.2.1. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the LCS/LCSD criteria. Use of a LCS pair in place of a MS/MSD must be documented.

9.3. Sample count

9.3.1. Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included. Additional MS/MSD sets are included in the sample count.

9.4. Method Blank

9.4.1. A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.4.2. Aqueous Method Blanks use 1000 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure.

9.4.3. Solid method blanks use approximately 30 g of sodium sulfate spiked with the surrogates. The method blank goes through the entire analytical procedure.

9.4.4. TCLP method blanks use 250 mL of leachate fluid spiked with the surrogates. SPLP method blanks use 1000 mL of leachate fluid spiked with the surrogates The leachate may optionally be diluted to 1000 mL with reagent water. The method blank goes through the entire analytical procedure.

9.5. Laboratory Control Sample (LCS)

9.5.1. Laboratory Control Samples are well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure.

9.5.2. The LCS is made up in the same way as the method blank (See sections 9.4.1 - 9.4.4) but spiked with the LCS standard and the surrogates.

9.6. Surrogates

9.6.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.6.2. Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.7.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike.

9.8. Initial Demonstration of Capability

9.8.1. The initial demonstration and method detection limit studies described in Section 13 must be acceptable before analysis of samples may begin.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into the appropriate size glass vial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ±0.01 mL. The "standard" glass vial is sealed, and the meniscus is marked by etching a line on the bottle. The glass vials containing the sample extracts are then compared against the "standard" glass vial to ensure the final volume is consistently 1.0 ± 0.01 mL. If a new box of glass vials are used, then the steps are repeated to further ensure that variations due to glass vial size and shape are minimized. A log is kept of the lot number of the glass vials and the day the glass vials were prepared.

11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (Section 11.2), continuous liquid/liquid extraction (Section 11.3), sonication extraction (Section 11.4), soxhlet extraction (Section 11.5), accelerated soxhlet (Section 11.6), waste dilution (Section 11.7), and extract concentration (Section 11.8).

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC Manager. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples.

11.2.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.2.2. Measure the initial sample pH by inserting a disposable pipette into the sample, and placing a drop of sample on the wide-range pH paper. Record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.2.3. Measure the initial volume using using the volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. The normal sample volume is 1 liter. Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for very dirty samples.

11.2.4. Prepare a method blank, LCS and/or LCSD, and MS/MSD for each batch as specified in Section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS and/or LCSD. The LCS and/or LCSD and MS/MSD are spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates.

11.2.5. Use 250 mL of leachate for TCLP pesticides and TCLP semivolatiles, measured in a beaker.

11.2.6. For a TCLP method blank, LCS and LCS Dup measure 250 mL of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Add 60 mL of methylene chloride to the separatory funnel. The TCLP leachate may be diluted to approximately 1 liter before extraction if desired.

11.2.7. Pour the sample into a separatory funnel. Add 60 mL of methylene chloride per sample. Place a labeled collection jar under each appropriate separatory funnel. Place a small amount of glass wool into a funnel and fill with anhydrous sodium sulfate. Place a funnel containing sodium sulfate on each collection jar. Spike the samples with surrogate and/or spike solutions.

11.2.8. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample by inserting a disposable pipette into the sample, and placing a drop of sample onto the pH paper. Record adjusted pH, spiking volumes, and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.2.9. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. An autoshaker may be used to shake and rotate the separatory funnel.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.10. Allow the organic layer to separate from the water phase until complete visible separation has been achieved. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends

upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in continuous liquid-liquid extraction (Section 11.3.). If this is done, the sample must be extracted as part of a valid CLLE batch.

***Note**: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

11.2.11. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water, replace the existing sodium sulfate with fresh drying agent.

11.2.12. Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.13. If extraction at a secondary pH is required, replace the filtration funnel and adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample onto the pH paper. Record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride, as outlined in Steps 11.2.7 to 11.2.10. Collect these three extracts in the same container used for the previous fraction.

11.2.14. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.15. Dispose of solvent and water remaining in the extractor into the appropriate waste container.

11.2.16. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.8 for concentration.

11.3. Continuous Liquid/Liquid Extraction from Water Samples.

11.3.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.3.2. Assemble the apparatus. Add approximately 250 mL of methylene chloride to the extractor body. Add 3 to 5 boiling chips to the round-bottom distilling flask. Label the flask with an extraction ID label.

11.3.3. Measure the initial sample pH with wide-range pH by inserting a disposable pipette into the sample, and placing a drop of sample onto the wide range pH paper. Record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log. Note on the benchsheet if there is any discrepancy.

11.3.4. Measure the initial volume using using the volume template. Place the template next to the sample bottle and read the volume marking from the template. Record this volume on the benchsheet. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS.

Use 250mL of leachate for TCLP for TCLP semivolatiles and TCLP pesticides. Use 1000 mL of leachate for SPLP semivolatiles and SPLP pesticides. Dilute to about 1 liter with reagent water.

For a TCLP method blank, LCS and LCS Dup measure 250 mL of the buffer solution used in the leaching procedure and transfer to the continuous liquid/liquid extractor. Dilute to about 1 liter with reagent water. For an SPLP method blank, LCS and LCS Dup measure 1000 mL of the buffer solution used in the leaching procedure and transfer to the continuous liquid/liquid extractor. No dilution with reagent water is required.

Less than one liter of sample may be used, for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event, dilute the sample to about 1 liter with reagent water. This must be documented with a Non-Conformance Memo.

11.3.5. Add reagent water to the extractor body until approximately 250 mL of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Prime the extractor using reagent water. The method blank and samples are spiked with the surrogates, the LCS and matrix spikes with the surrogates and matrix spiking solutions.

Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible. Attach cold condenser (about 10°C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours. (24 hours required for 600 series)

If extraction at a secondary pH is required, (see Table 1) turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure by inserting a disposable pipette into the sample, and placing a drop of sample on the pH paper. Record the adjusted

pH on the benchsheet. Reattach the condenser and turn on heating mantle. Extract for 18-24 hours.

Turn off the heating mantle and allow the extractor to cool.

Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.8 for concentration.

11.4. Sonication

11.4.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.4.2. Decant and discard any water layer on a sediment/soil sample, unless there are specific instructions not to decant the water. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.4.3. Weigh 30 g of sample ± 0.2g into a 250 or 400 mL beaker or wide-mouth jar. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet.

11.4.4. Add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (See Table 4). Add the appropriate volume of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes (see Table 3 for appropriate amounts). Refer to Table 6 for details of the surrogate spiking solutions. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

11.4.5. Mix the weighed and spiked/surrogated sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.6. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP. Use 30 g of sodium sulfate for the method blank and LCS. Use 30 g ± 0.2g each, of parent sample for the MS and MSD samples.

11.4.7. Immediately add a minimum of 100 mL of solvent to the beaker.

Solvents:

Semivolatile GC/MS, TPH, Organochlorine pesticides and PCBs	1:1 v/v Methylene Chloride / Acetone

Note: Steps 11.4.5 - 11.4.9 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.8. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.9. Sonicate for three minutes, making sure the entire sample is agitated.
Note: Do ***not*** use ***Microtip*** probe.

11.4.10. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.4.11. Place the prepared funnel on a labeled collection apparatus (beaker or K-D Apparatus).

11.4.12. Decant and filter extracts through the prepared funnel into the collection apparatus. a clean beaker or K-D Apparatus.

11.4.13. Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone appropriate solvent (Refer to Table in 11.4.7).

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.4.14. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.8 for concentration

11.4.15. Sonicator Tuning: Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned quarterly and if a new horn is installed.

11.5. Soxhlet

11.5.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.5.2. Decant and discard any water layer on a sediment/soil sample, unless there are specific instructions not to decant. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in a clean beaker and

homogenize. Upon completion of homogenization in the beaker, return the sample to original container. Discard foreign objects such as sticks, leaves, and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials, consult with the client (via the Project manager or Administrator).

11.5.3. Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Weigh 30 g of sample ± 0.2g into a thimble or in a beaker, recording the weight to the nearest 0.01 g on the benchsheet.. Sample weights less than 30 g but over 5 g may be used if the appropriate reporting limits can be met.

11.5.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix for the LCS. The parent sample is used for the MS/MSD. The weight of sodium sulfate used should be approximately the weight of soil used in each sample.

11.5.5. Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 3, 4, 5, and 6.

11.5.6. Add anhydrous sodium sulfate to each sample and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet extractor thimble, but do not pack the thimble tightly. The Soxhlet extractor or extraction thimble must drain freely for the duration of the extraction period. A glass wool plug below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

11.5.7. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

Note: If a reduced quantity of sample is extracted, it is usually necessary to increase the amount of sodium sulfate added or increase the solvent boiling rate to properly set the cycling rate.

Solvents:

Semivolatile GC/MS, OPP, PAH, TPH Organochlorine pesticides and PCBs	1:1 v/v Methylene Chloride / Acetone

11.5.8. Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask.

11.5.9. The sample is now ready for the concentration step (Section 11.8).

11.5.10 Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.8 for concentration.

11.6. Accelerated Soxhlet (Soxtherm Trade Name)

11.6.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.6.2. Decant and discard any water layer on a sediment/soil sample, unless there are specific instructions not to decant. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker, return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials, consult with the client (via the Project Manager or Administrator).

11.6.3. Weigh 30 g of sample ± 0.2 g into a beaker or wide-mouth jar, recording the weight to the nearest 0.01 g on the benchsheet. Add 30 g of anhydrous sodium sulfate to each sample, and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to an accelerated soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug below the sample in the thimble is required.

11.6.4. Sample weights less than 30 g, but over 5 g may be used if the appropriate reporting limits can be met.

11.6.5. Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix. The weight of sodium sulfate used should be approximately the weight of soil used in each sample.

11.6.6. Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 3, 4, 5, and 6.

11.6.7. Place thimble in the extract beaker containing at least six clean boiling chips and add approximately 110 mL of solvent. Place beakers into positions on the accelerated soxhlet unit. Run appropriate program for the extraction solvent. Check the system for leaks at the joints periodically.

Solvents:

Semivolatile MS, PAH, TPH Semivolatile GC PCB, PEST, OPP	1:1 v/v Methylene Chloride / Acetone 1:1 v/v Hexane/Acetone

11.6.8. Upon completion of the program, remove the extract beaker from the unit, let cool, and dispose of the extracted sample.

11.6.9. Transfer extract into a culture tube, rinsing the extract beaker to complete the

quantitative transfer. Rinse the extractor beaker, which contained the solvent extract with 10-15 mL of the appropriate solvent and pour it through the sodium sulfate drying funnel. Rinse the funnel with 10-15 mL of methylene chloride to complete the quantitative transfer.

11.6.10. Place culture tubes on nitrogen evap and reduce to approximately 1-2mL and add appropriate solvent according to the extraction performed to bring the sample to the correct final volume.

11.7. Waste Dilution

11.7.1. Remove surrogate and matrix spiking solutions from refrigerator and allow to return to room temperature.

11.7.2. Label the vial with the sample number. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest ±0.01 g.

11.7.3. For the blank and LCS/LCSD, add a small amount of the appropriate solvent to the vial. Add appropriate volume of surrogate and spike solutions (Table 3).

11.7.4. Dilute to 10 mL with the appropriate solvent. (Methylene Chloride for GC/MS Semi and GCS TPH. Add 10 mL of appropriate solvent (Hexane) for GCS pesticide and/or PCB analysis. 8015B waste dilutions are diluted to approximately 10mL with DCM and are placed on the nitrogen evaporation unit to reduce to a 2 mL final volume.

11.7.5. Cap and shake or vortex each extract.

11.7.6. The sample is now ready for analysis.

11.8 Concentration

According to the type of sample, different solvents and final volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.8.1. Kuderna-Danish (KD) Method:

11.8.1.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Label the CT and KD. Transfer the sample to the labeled K-D flask, filtering Continuous Liquid/Liquid and Soxhlet samples through funnels filled with sodium sulfate. Rinse the funnel with 20-30 mL of methylene chloride to complete the quantitative transfer.

11.8.1.2. Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three-ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column. (This

is important to ensure that the balls are not stuck, and the column will work properly). Attach to the KD flask.

11.8.1.3 Place the KD apparatus on a water bath (90-98°C) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood.

11.8.1.4 Concentrate to 15-20 mL. If the determinative method requires a solvent exchange add the appropriate exchange solvent to the top of the Snyder Column, and then continue the water bath concentration back down to 5-8 mL. Refer to Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.8.1.5. Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.9. Nitrogen Evaporation to Final Concentration

11.9.1. Transfer the CT to the evaporation apparatus

11.9.2. Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

Note: It is very important not to concentrate to dryness as analytes will be lost.
Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The procedure for the determination of the method detection limit is given in TestAmerica North Canton QA Policy S-Q-003 and NC-QA-0021.

13.2. Initial Demonstration

13.2.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The spiking level should be equivalent to a mid-level calibration. (For certain tests more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.2.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

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

15.2.1. Extracted aqueous samples contaminated with methylene chloride. This tank is then periodically rolled to the tank room, the pH is verified, the contents are neutralized with sodium bicarbonate, the pH re-verified and the Dichloromethane waste drained into a waste drum located outside the building. The waste water is discharged to the POTW

15.2.2. Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride/acetone or acetone/hexane from the extract drying step. These materials are disposed of in the solid waste and debris in a red container located in the extractions lab.

15.2.3. **Assorted flammable solvent waste from various rinses**. These wastes are put into the halogenated/non-halogenated 25 gallon solvent waste container located under the fume hood in extractions.

15.2.4. **Methylene chloride waste from various rinses:** These wastes are disposed of in the liquid-liquid separation unit.

15.2.5. **Hexane- Hexane waste:** These samples are to be disposed in the flammable waste.

15.2.6. **Waste Hexane in vials.** These vials are placed in the vial waste located in the GC prep laboratory.

15.2.7. **Waste Methylene Chloride sample vials**. These vials are placed in the vial waste located in the GC prep laboratory.

15.2.8. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane.** These materials are disposed of in the solid waste and debris in a red container located in the extractions lab.

15.2.9. Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCB's must be segregated into their own waste stream. PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB and PCB vial waste.

15.2.10. Solvent Recovery System Waste. Methylene Chloride waste from the Solvent Recovery System is collected and disposed of in the liquid-liquid separation unit. Acetone/Methylene Chloride waste from this system is disposed of in the flammable waste containers located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd

Edition, Final Update III (December 1996). Sections 3500B, 3510C, 3520C, 3540C, 3550B, 3600C, 3610B, 3620B, 3640A, 3650B, 3660B, AND 3665A.

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018, current version.

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021, current version.

16.2.5. Supplemental Practices for DoD Project Work SOP, NC-QA-0016

16.2.6. Method Detection Limit Studies, CA-Q-S-006, current version.

16.2.7. CORP-GC-0001NC, Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8082, 8151A, 8015B, and 615, current version.

16.2.8. CORP-MS-0001NC, GC/MS Analysis based on Method 8270C, current version.

16.2.9. NC-GC-0007, Analysis of Pesticides and PCBs by EPA Method 608, current version.

16.2.10. NC-MS-0003, GC/MS Semivolatile Organic Compounds Capillary Column Technique Based on EPA Method 625, current version.

16.2.11. Standards and Reagents, SOP NC-QA-0017.

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.2. Tables

TABLE 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA: 8270C[1]	1-2 (acid first) or 11-12 (base first)	11-12 (base first) or 1-2 (acid first)
625	11-12 (base first) or 1-2 (acid first)	1-2 (acid first) or 11-12 (base first)
Pest/PCB: 8081A, 8082 and 608	5-9	None
Hydrocarbons: 8015B	As received	None

[1] If the laboratory has validated acid only 8270 extraction for the target compound list required then the base extraction step may be omitted. The required validation consists of a 4 replicate initial demonstration of capability and a method detection limit study. (See Section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

TABLE 2
Final Volumes and Exchange Solvents

Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	2.0 mL
PCB	Approximately 18 mL Hexane – water Approximately 36 mL Hexane - solid	10.0 for solids 5.0 for H2O 2.0 for H2O*
Pesticides	Approximately 18 mL Hexane	10.0 for solids 5.0 for H2O
Pesticides/TCLP	Approximately 18 mL Hexane	3.0 mL
BNA – SIM	N/A	2.0 mL - Solids & H2O
TPH	N/A	1.0

* Michigan work requires a final volume of 2 mL.

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

TABLE 3

Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.2
BNA / SIM	100/150 ppm BNA	0.2 / 0.02
BNA Waste Dilution	100/150 ppm BNA	0.5
PEST	0.2 ppm DCB/TCX	1.0
TPH	40ng Nonane (C9)	1.0
PCB	0.2 ppm DCB/TCX	1.0

TABLE 4
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	100 ppm BNA All-Analyte Spike and Restek Spike	0.2
	Waste Dilution	0.5
BNA / SIM	100 ppm BNA All-Analyte Spike and Restek Spike	0.2 / 0.02
PEST	Pest NPDES Spike	1.0
PEST TCLP	Pest TCLP Spike	1.0
PCB	10 ppm PCB Spike	1.0
TPH	See Spike List – Table 6	1.0

TABLE 5

Surrogate Spike Components

Analyte Group	Compounds	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	100
	Nitrobenzene-d5	100
	p-Terphenyl-d14	100
	2-Fluorophenol	150
	Phenol-d6	150
	2,4,6-Tribromophenol	150
	1,2-Dichlorobenzene-d4	100
	2-Chlorophenol-d4	150
PEST	Decachlorobiphenyl	0.2
PCB	Tetrachloro-m-xylene	0.2
TPH	Nonane (C9)	40.0

TABLE 6
Matrix Spike Components

Type	Compounds	Conc. (µg/mL)
BNA	Acenaphthene	100
	4-Chloro-3-Methylphenol	150
	2-Chlorophenol	150
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	4-Nitrophenol	150
	N-Nitroso-Di-n-Propylamine	100
	Pentachlorophenol	150
	Phenol	150
	Pyrene	100
BNA	1,2,4-Trichlorobenzene	100

Type	Compounds	Conc. (µg/mL)
	1,4-Dichlorobenzene	100
	2,4-Dinitrotoluene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	2-Methylphenol	100
	3-Methylphenol	100
	4-Methylphenol	100
	Nitrobenzene	100
	Pentachlorophenol	100
	Pyridine	100
	2,4,5-Trichlorophenol	100
	2,4,6-Trichlorophenol	100
	Acenaphthene	100
	Acenaphthylene	100
	Anthracene	100
	Benzo(a)anthracene	100
	Benzo(b)fluoranthene	100
	Benzo(k)fluoranthene	100
	Benzo(a)pyrene	100
	Benzo(ghi)perylene	100
	Benzyl butyl phthalate	100
	Bis(2-chloroethyl)ether	100
	Bis(2-chloroethoxy)methane	100
	Bis(2-ethylhexyl)phthalate	100
	Bis(2-chloroisopropyl)ether	100
	4-Bromophenyl phenyl ether	100
BNA	2-Chloronaphthalene	100

Type	Compounds	Conc. (µg/mL)
	4-Chlorophenyl phenyl ether	100
	Chrysene	100
	Dibenzo(a,h)anthracene	100
	Di-n-butylphthalate	100
	1,3-Dichlorobenzene	100
	1,2-Dichlorobenzene	100
	1,4-Dichlorobenzene	100
	3,3'-Dichlorobenzidine	100
	Diethyl phthalate	100
	Dimethyl phthalate	100
	2,4-Dinitrotoluene	100
	2,6-Dinitrotoluene	100
	Di-n-octylphthalate	100
	Fluoranthene	100
	Fluorene	100
	Hexachlorobenzene	100
	Hexachlorobutadiene	100
	Hexachloroethane	100
	Indeno(1,2,3-cd)pyrene	100
	Isophorone	100
	Naphthalene	100
	Nitrobenzene	100
	N-Nitrosodi-n-propylamine	100
	Phenanthrene	100
	Pyrene	100
	1,2,4-Trichlorobenzene	100
	4-Chloro-3-methylphenol	100
BNA	2-Chlorophenol	100

Type	Compounds	Conc. (µg/mL)
	2,4-Dichlorophenol	100
	2,4-Dimethylphenol	100
	2,4-Dinitrophenol	100
	2-Methyl-4,6-dinitrophenol	100
	2-Nitrophenol	100
	4-Nitrophenol	100
	Pentachlorophenol	100
	Phenol	100
	2,4,6-Trichlorophenol	100
	Acetophenone	100
	Atrazine	100
	Caprolactum	100
	Benzaldehyde	100
	1,1′-Biphenyl	100
	Safrole	100
	1,4-Dioxane	100
	Pronamide	100
	p-Chlorobenzilate	100
	Phenacetin	100
	Ethyl methanesulfonate	100
	2-Picoline	100
	Phorate	100
	Quinoline	100

Type	Compounds	Conc. (µg/mL)
Pest TCLP	Heptachlor	0.5
	Heptachlor epoxide	0.5
	Lindane	0.5
	Endrin	0.5
	Methoxychlor	1.0
Pest NPDES/Pest	Alrin	1.0
	Alpha-BHC	1.0
	beta-BHC	1.0
	delta-BHC	1.0
	gamma-BHC (Lindane)	1.0
	4,4'-DDD	1.0
	4,4'-DDE	1.0
	4,4'-DDT	1.0
	Dieldrin	1.0
	alpha-Endosulfan	1.0
	beta-Endosulfan	1.0
	Endosulfan Sulfate	1.0
	Endrin	1.0
	Heptachlor	1.0
	Heptachlor Epoxide	1.0

Diesel Range Organics (8015B) Spike	
Compound	**Final Concentration**
n-decane	50 µg/ml
n-dodecane	50 µg/ml
n-tetradecane	50 µg/ml
n-hexadecane	50 µg/ml
n-octadecane	50 µg/ml
n-eicosane	50 µg/ml
n-docosane	50 µg/ml
n-tetracosane	50 µg/ml
n-hexacosane	50 µg/ml
n-octacosane	50 µg/ml

TESTAMERICA NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: **EXTRACTION PROCEDURE FOR CHLORINATED ACID HERBICIDES BASED ON METHOD 8151A**

(SUPERSEDES: Revision 0, Dated 10/17/06)

Reviewed by:	Technology Specialist	9-24-07 Date
Approved by:	Quality Assurance Manager	9/24/07 Date
Approved by:	Environmental Health and Safety Coordinator	9-24-07 Date
Approved by:	Laboratory Director	9/25/07 Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the extraction of chlorinated herbicides in waters, solids, oils, and TCLP extracts. Appropriate compounds for extraction by this method are listed in CORP-GC-0001.

1.2. The applicable LIMS method code is QS (8151A).

2. SUMMARY OF METHOD

2.1. This method is based on SW846 method 8151A. Aqueous samples are hydrolyzed if esters and acids are to be determined, then washed with methylene chloride by a separatory funnel extraction. After acidifying the sample the free acids are extracted into diethyl ether.

2.2. Solids are extracted into methylene chloride/ acetone by sonication. If esters and acids are to be determined, the extract is hydrolyzed and extracted into diethyl ether.

2.3. For both soils and aqueous samples, the free acid herbicides in the ether extract are esterified. The final volume is adjusted to prepare the extract for gas chromatography.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the TestAmerica North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.3 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. The determination using pentafluorobenzylation is more sensitive, and more prone to interferences from the presence of organic acids or phenols than by methylation.

4.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.

4.5 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

4.6 Sample extracts should be dry prior to methylation or else poor recoveries will be obtained.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Lab Specific Addendum to the CSM, and this document.

5.2 DIAZOMETHANE is an extremely toxic gas with an explosion potential. Since the explosion potential is catalyzed by imperfections in glass, generation of diazomethane must be carried out in glassware free of scratches, cracks, chips, and which does not have ground glass joints. Solutions of diazomethane will be kept at temperatures below 90°C. Diazomethane must be generated and handled in a fume hood.

5.3 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
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 2 Mg/M^3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. 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 (1)	Corrosive Oxidizer Dehydrator Poison Carcinogen	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. 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.
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 within six months.**
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 Hydroxide	Corrosive	2 Mg/M^3-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.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e.g., pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.

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

5.4. Eye protection that protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit. If more than 500 mL of Methylene chloride is spilled, evacuate the area until the area has been cleaned by EH&S.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to a laboratory supervisor and the EH&S Coordinator.

5.8. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus and glass separatory funnels have ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least two hours. Alternatively the glassware can be solvent rinsed with acetone or methanol followed by methylene chloride after the water rinse

6.2. Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Sep fun.	Soni	Conc
Separatory Funnel: 2 L	√		
Separatory Funnel Rack	√		
pH indicator paper, wide-range: covers extraction pH	√	√	
Graduated cylinder: 1 liter. (other sizes may be used)	√		
Centrifuge	√	√	
Auto-Shaker	√		
Methylene Chloride Collection Tank	√	√	√
Solvent Dispenser Pump or 100 mL Graduated Cylinder	√	√	√
Beakers: 250 & 400 mL, graduated		√	
450mL wide-mouth glass jars		√	
1 L amber wide mouth glass jars		√	
Balance: >100 g capacity, accurate ±0.1 g		√	
Sonicator (at least 300 watts)		√	
Sonicator horn, 3/4 inch		√	
Kuderna-Danish (K-D) Apparatus: 500 mL			√
Concentrator Tube: 10 mL, attached to K-D with clips			√
Snyder Column: Three-ball macro			√
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.			√
Vials: Glass 10 mL capacity with Teflon®-lined screw-cap			√
Nitrogen Blowdown Apparatus			√
Nitrogen: Reagent grade.			√
Culture tubes: 10 mL, 16 mmx100 mm			√
Syringe: 1 mL or positive displacement pipette	√	√	
Glass Wool	√	√	
Glass Funnel: 75 X 75 mm	√	√	√
Disposable Pipettes	√	√	√
Aluminum foil	√	√	√
Paper Towels	√	√	√
Balance: >1400 g capacity, accurate ±.1 g;	√	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep fun.	Soni	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	√		
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√		
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	√	√	
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	√	
Hydrochloric Acid (HCl)		√	
Organic free reagent water.	√		
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√	√
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, toluene, pesticide quality or equivalent	√	√	√
Acetone: Used for cleaning	√	√	√

7.1.1. Potassium hydroxide solution, 37% aqueous solution, (w/v): Dissolve 37 g of potassium hydroxide pellets in reagent water and dilute to 100 mL. **Caution:** Considerable heat will be generated. Other volumes of solution may be made up as convenient, in the same proportions.

7.1.2. Toluene, reagent grade.

7.1.3. Sodium sulfate (Na2SO4), anhydrous, granular, acidified: Use approximately 2000g of oven muffled Na2SO4 create a slurry using enough diethyl ether to cover. Add approximately 80mLs of concentrated H2SO4, mix thoroughly. Place the mixture on steam bath in hood to allow ether to evaporate. Larger or smaller batches may be created using the same reagents in the same proportions. Store in a desiccator. Check the pH of the reagent prior to use by mixing 1 g of the soidum sulfate with 5mL of reagent grade water and testing the pH. The pH must be ≤ 4.

7.1.4. Sodium Chloride, NaCl

7.1.5. BF3-Methanol, Boron trifluoride-MeOH, lab use only

7.1.6. Diethyl ether, reagent grade

7.1.6.1. Diethyl ether used for this procedure should be stabilized with BHT, not with ethanol, as when ethanol-stabilized ether is used, the methylation reaction may not proceed efficiently, leading to low recoveries of target analytes.

7.1.7. Trimethylsilydiaomethane

7.1.8. Methanol, reagent grade.

7.1.9. Silica gel

7.1 Standards

7.1.1 Stock Standards

Stock standards are purchased as certified solutions or prepared from neats. Semivolatile stock standards are stored per manufacturer instructions. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in-house, or from the time the ampoule is opened if purchased). Standards must be allowed to come to room temperature before use.

7.2.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be stored per manufacturer instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.3. Matrix Spiking and Laboratory Control Spiking Standards.

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards. Spiking solutions must be stored per manufacturer instructions. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.2.4. Surrogate Standard. See Table 1.

7.2.5. Matrix Spike and LCS standard. See Table 2.

8 SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1 Samples are not chemically preserved.

8.2 Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps and protected from light.

8.3 Holding Times

8.3.1 Extraction is initiated within seven days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2 For TCLP leachates, extraction is initiated within seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step. If the filtration step requires extended times, this time counts as part of the seven-day holding time.

8.3.3 Analysis of the extracts is completed within 40 days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

9.1.1. The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike / matrix spike duplicate. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See Policy QA-003 for further definition of the batch.

9.2. Insufficient Sample

9.2.1. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the LCS/LCSD criteria. Use of an LCS pair in place of an MS/MSD must be documented.

9.3. Method Blank

9.3.1. A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.3.2. Aqueous Method Blanks use 500 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.3.3. Solid method blanks use approximately 50 g of sodium sulfate spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.3.4. TCLP method blanks use 100mL of leachate fluid spiked with the surrogates. The leachate may optionally be diluted to 500 mL with reagent water. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.4. Laboratory Control Sample (LCS)

9.4.1. Laboratory Control Samples are well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.4.2. The LCS is made up in the same way as the method blank (See sections 9.3.1 - 9.3.4), but spiked with the LCS standard and the surrogates.

9.5. Surrogates

9.5.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.5.2. Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure the appropriate volume of solvent into an autovial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ±0.01 mL. The "standard" autovial is sealed and the top and bottom of the meniscus are marked. The autovials containing the sample extracts are then compared against the "standard" vial to ensure that the final volume is consistently 1.0 ± 0.01 mL. If a new box of autovials are used, then the steps are repeated to further ensure that variations

due to vial size and shape are minimized. A log is kept to track the day the vials were prepared.

11. PROCEDURE

11.1 Preparation of Aqueous Samples

11.1.1 Remove surrogate and matrix spiking solutions from refrigerator, and allow to return to room temperature.

11.1.2 Volumetrically transfer 500 mLs into a 1 liter wide-mouth amber jar. For TCLP samples volumetrically transfer 100mLs of sample in to jar and dilute to 500mLs with reagent grade water.

11.1.3 Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to Tables 1 and 2)

11.1.4 Add 125-150g of NaCl to samples and QC Samples and shake to dissolve the salt.

11.1.5 Hydrolysis

11.1.5.1 Add approximately 3 mL of 10N NaOH to the sample, stir sample, and check the pH of the sample with pH paper. If the pH of the sample is not $\geq$12 adjust to $\geq$12 by adding more NaOH.

11.1.5.2 Add 300 mL of methylene chloride to the amber jar.

11.1.5.3 Prior to placing samples in tumbler, the samples should be shaken or rotated vigorously for two minutes, venting as necessary. Place the samples in tumbler and allow them to tumble for one hour.

11.1.5.4 Pour content of amber jar into a pre-rinsed Teflon sep funnel, let stand for 10 minutes to allow organic layer to completely settle. If an emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

11.1.5.5 Discard the methylene chloride phase.

11.1.6 Extraction of Acids

11.1.6.1 Add 4-8 mL of 1:1 sulfuric acid to the sample. Seal, and shake to mix. Check the pH of the sample with pH paper. If the pH is not ≤ 2, and more acid to adjust the pH to ≤ 2. ***Caution: Addition of acid may cause heat and / or pressure build up.***

11.1.6.2 Add 100mLs of diethyl ether to sample and extract serially three times for two minutes by auto rotator, venting as necessary. Allow organic layer to separate from aqueous layer by allowing sample to sit for ten minutes prior to collecting ether layer. Drain aqueous layer into amber liter jar, and collect ether layer in a mason jar containing approximately 20g of acidified anhydrous sodium sulfate.

11.1.6.3 Return the aqueous phase to the separatory funnel, add 100 mL diethyl ether and repeat the extraction procedure a second time, combining the ether extracts. Repeat the extraction a third time with 100 mL diethyl ether. Discard the aqueous phase after the third extraction.

11.1.6.4 Allow the extract to remain in contact with the sodium sulfate for at least two hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken.

11.1.6.5 Proceed to Section 11.5, Concentration.

11.2 Extraction of soil and sediment samples

11.2.1 Remove surrogate and matrix spiking solutions from refrigerator, and allow to return to room temperature.

11.2.2 Decant and discard any water layer on a sediment/soil sample. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible, place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker, return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials, consult with the Project Manager.

11.2.3 Weigh 50.0 g ($\pm$ 0.20g) of moist solid sample into a clean glass jar. Use 50 g of sodium sulfate for the Method Blank and the LCS. Acidify the sample with approximately 5 mL of concentrated HCl.

11.2.4 There should be a small amount of liquid phase. Stir well with a spatula. (Note: This is not necessary for the method blank or LCS.)

11.2.5 Stir with a spatula and check the pH of the liquid phase. Add more acid if necessary to bring the pH to <2, repeating the stirring after each acid addition.

11.2.6 Dry samples with oven muffled sodium sulfate until the sample is free flowing. The pH of the sodium sulfate should be checked prior to use by taking a few grams and adding to reagent water. The pH should be $\leq$ 7. If not, acidified anhydrous sodium sulfate should be used.

11.2.7 Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to Tables 1 and 2)

11.2.8 Add a minimum of 100 mL of 1:1 methylene chloride:acetone to the beaker.

11.2.9 Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.2.10 Sonicate for three minutes, making sure the entire sample is agitated.

11.2.11 Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.2.12 Place the prepared funnel on a collection apparatus. If the herbicide esters are to be determined (normally the case), the collection apparatus is glassware suitable for the hydrolysis step, typically a KD flask or Turbovap tube.

11.2.13 Decant and filter extracts through the prepared funnel into the collection apparatus.

11.2.14 Repeat the extraction two more times with additional 100 mL minimum portions of Methylene Chloride each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.3 Hydrolysis

11.3.1 Add 5 mL of 37% aqueous potassium hydroxide and 30 mL of reagent grade water to the extract. Shake the sample vigorously for 30 seconds and let stand for 10 minutes. Check the pH with pH paper. If the pH is not ≥12, adjust with additional KOH.

11.3.2 Heat on a water bath at 94±4°C until the organic layer is completely evaporated and the Synder column has stopped chattering.

11.3.3 Before transferring the extract to a separatory funnel assure pH is still ≥12, if not, adjust by adding more 37%KOH and 50mL of Methylene Chloride and return to steam bath, for 15 minutes, and verify the pH is ≥12 when finished.

11.3.4 Transfer the solution to a separatory funnel and extract serially three times with 100 mL portions of methylene chloride. **Discard the methylene chloride phase.** After the third time, let stand for 10 minutes prior to discarding the last layer of Methylene Chloride to ensure all Methylene Chloride is removed from the aqueous layer. The aqueous layer will contain the herbicides as long as the aqueous layer remains basic. If an emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation

11.4 Extraction of Acids

11.4.1 Add 4-8 mL of 1:1 sulfuric acid to the sample. Seal, and shake to mix. Check the pH of the sample with pH paper. If the pH is not ≤2, add more acid to adjust the pH to ≤ 2. ***Caution: Addition of acid may cause heat and / or pressure build-up.***

11.4.2 Add 100mLs of diethyl ether to sample and extract serially three times for two minutes by auto rotator, venting as necessary. Allow organic layer to separate from aqueous layer by allowing sample to sit for 10 minutes prior to collecting ether layer. Drain aqueous layer into amber liter jar, and collect ether layer in a mason jar containing approximately 20g of acidified anhydrous sodium sulfate.

11.4.3 Return the aqueous phase to the separatory funnel, add 100 mL diethyl ether and repeat the extraction procedure a second time, combining the ether extracts. Repeat the extraction a third time with 100 mL diethyl ether. Discard the aqueous phase after the third extraction.

11.4.4 Allow the extract to remain in contact with the sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium

sulfate. The amount of sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken.

11.4.5 Proceed to Section 11.5, Concentration.

11.5 Concentration

11.5.1 Transfer the ether extract into a 500 mL K-D flask **equipped with a 10 mL concentrator tube.** Crush the caked sodium sulfate during transfer. Rinse the flask or bottle with 20-30 mL ether to complete transfer.

11.5.2 Attach a three-ball Snyder column to the K-D apparatus, pre-wet the column with a few mL of ether from the top, and place the apparatus on a water bath at approximately 65°C. At the proper rate of distillation, the balls of the column will chatter, but the chambers will not flood. When the apparent volume reaches approximately 20 mL, exchange with approximately 18mL of Hexane, remove from the water bath and allow to completely cool.

Note: TCLP extracts are exchanged with 4 mL of Toluene. When the apparent volume reaches approximately 6 mL, remove from the steam bath and allow to cool completely.

11.5.3 Carefully disassemble the concentrator tube and rinse the lower glass joint with a small amount of diethyl ether.

11.5.4 Place the extracts on the Nitrogen Blowdown and allow to concentrate to 2 mL. **Note:** For Non-TCLP extracts, the extracts should be quantitatively transferred to a test tube prior to being placed on the blowdown.

11.5.5 The extract is now ready for esterification by Trimethylsilydiazomethane (Section 11.7) or the TCLP esterification by Boron Triflouride Method (Section 11.6).

11.6 Esterification by Boron trifluoride (TCLP extracts only)

11.6.1 To the concentrator tube with the extract, add approximately 2 mL of Boron trifluoride. Place a three-ball micro-Snyder column on the concentrator tube and place in the Hot-Block water bath adjusted to 35-60°C for 60 minutes. Remove and let cool for approximately ten minutes.

11.6.2 With a 10 mL graduated disposable 5-3/4" pipette 4.5 mL of 5% neutral sodium sulfate and place it in the concentrator tube. Seal with a tight fitting ground glass stopper. Vortex the mixture for one minute. Let stand for ten minutes to settle. With a 5-3/4" disposable pipette, withdraw the bottom aqueous layer into a 16 x

100 culture tube for proper disposal.

11.6.3 Prepare a clean up column in a 5-3/4" disposable pipette by placing a small amount of glass wool in the narrow end of the pipette and add about ½ inch – 1 inch of florisil and sodium sulfate each. Leave about a one inch gap at the top. Place the extract in the clean-up column and gently force it through by using a pipette bulb into a small test tube. **Care should be taken to avoid channeling.** Rinse the concentrator tube with 1-2 mL of toluene. Transfer the column rinsate into the test tube. Rinse the column with additional toluene. There should be approximately 4 mL collected in the test tube. Bring the final volume to 10 mL with toluene by visually comparing it to a calibrated collection tube. **Note:** It is critical that all toluene is retained and no water should enter the column.

11.7 Esterification by Trimethylsilydiazomethane

11.7.1 Add 200 uL of methanol to the extract, followed by 200 uL of trimethyldiazomethane solution. The extract will turn a yellow color. If this does not occur, add additional 200 uL aliquots of trimethyldiazomethane solution until the yellow color persists. Check the sample every 15 minutes for yellow color. If the yellow disappears in this time frame, add additional 200 uL aliquots of trimethyldiazomethane solution until the yellow color persists.

11.7.2 Allow the extract to stand for at least one hour at room temperature to allow the methylation reaction to occur. The reaction is halted with the addition of salicic acid. The extract is then brought up to a final volume of 10mLs with hexane and submitted for analysis.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The procedure for the determination of the method detection limit is given in TestAmerica North Canton QA Policy S-Q-003.

13.2. Initial Demonstration

13.2.1. Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The spiking level should be equivalent to a mid-level calibration. (For certain tests more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.2.2. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.3. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modification that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

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

15.2.1. **Aqueous acidic waste.** These wastes are disposed of in the liquid-liquid separation unit.

15.2.2. **Non-hazardous sodium sulfate**. Non hazardous substances can be disposed of in the regular trash.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Chlorinated Herbicides, Method 8151A.

16.1.2. Corporate Quality Management Plan (QMP), current version.

16.1.3. TestAmerica Laboratory Quality Manual (LQM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018, current version.

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021, current version.

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.6. Method Detection Limit Studies, S-Q-003, current version.

16.2.7. CORP-GC-0001NC, Gas Chromatographic Analysis based on Method 8000B, 8021B, 8081A, 8082, 8151A, 8310, 8015B, and 615, current version.

16.2.8 Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Section 7.5 of Method 8151A lists Diazomethane and PFB for the esterification process. The laboratory is using Trimethylsilyldiazomethane and Boron Trifluoride for the esterification process.

17.2. Tables

Table 1
Herbicide Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
Herbicides	Herbicides TCLP	1.0
Herbicides	Herbicides Soil & Water	1.0

Table 2
Herbicide Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
Herbicides	Herbicides EA	1.0
Herbicides	Herbicides MS-TCLP	1.0

Table 3
Herbicide Surrogate Spike Components

Type	Compounds	Solvent	Conc (ug/mL)
Herbicides WS	2,4-DCAA	Acetone	2
Herbicides SS	2,4-DCAA	Acetone Methanol	20

[1]The surrogate is spiked as the free acid

Table 4

Herbicide Matrix Spike Components

Type	Compounds[1]	Compounds[1] Solvent	EA Conc. (ug/mL)	TCLP Conc. (ug/mL)
Herbicides MS	2,4-D	Methanol	16	2
	2,4-DB		16	2
	2,4,5-TP (Silvex)		4	0.5
	Dalapon		8	1
	Dicamba		8	1
	Dichloroprop		16	1
	Dinoseb		2.5	0.3
	2,4,5-T		4	0.5
	MCPA		1600	
	MCPP		1600	
	Pentachlorophenol		2	

[1]The herbicide spiking solution contains the herbicides as the free acids.

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. CORP-MS-0001NC, Rev. 2.12
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Cover Page

Title: GC/MS Analysis Based
[Method: **Method 8270C - UPDATE III**]

Approvals (Signature/Date):

Technology Specialist	Date 12/14/07	Health & Safety Coordinator	Date 12-14-07
Quality Assurance Manager	Date 12/13/07	Laboratory Director	Date 12/14/07
Technical Director	Date 12/14/07		

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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C

(SUPERSEDES: Revision 2.11, Dated 02/03/06)

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1. SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in Section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3 The standard reporting limit of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.4 The associated LIMS code is QL (8270C).

2. SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel, and/or a continuous extractor. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or pressurized fluid extraction. The extract is dried, concentrated to a final volume of 2 mL for waters and soils, and analyzed by GC/MS. Extraction procedures are detailed in SOP# CORP-OP-0001NC. Qualitative identification of

the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a

single characteristic ion.

3. DEFINITIONS

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the STL North Canton QC Program document (QA-003) for further details of the batch definition.

3.4 Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5 LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6 MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method for the matrix by measuring relative percent difference.

4. INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5 SAFETY PRECAUTIONS

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine.

5.4 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
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.			

5.5 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

5.6 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.7 It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.8 Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.9 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported immediately to a laboratory supervisor and the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 20m x 0.18mm ID, 0.36µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when the GC/MS tuning standard is injected through the GC.

6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6 Syringe: 5 µL Hamilton Laboratory grade syringes or equivalent.

6.7 Carrier gas: Ultra high purity helium.

7. REAGENTS AND STANDARDS

7.1 A minimum five point calibration curve is prepared. If a quadratic regression is used, six points must be analyzed for the calibration curve. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An Internal Standard solution is prepared by diluting a purchased standard. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard. All components are at 25 ug/mL.

7.5 The standards listed in 7.1 to 7.4 should be refrigerated at $\leq 6^{\circ}C$ when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year.

8. SAMPLE PRESERVATION AND STORAGE

8.1 Sample extracts are stored at $4 \pm 2^{\circ}C$. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will be stored for 30 days after invoicing.)

8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9. QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2 For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2 Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined periodically. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, MS and LCS. Precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference +/- 3 standard deviations. Control limits are established by the laboratory as described in SOP NC-QA-0018. Control limits are easily accessible via the LIMs (QC Browser program).

9.2.2 If samples are diluted, the surrogate and matrix spike recoveries will be reported with a DIL flag. Any analyte outside of the control limits will be flagged with JDIL. For DoD projects all surrogates must be within control limits.

9.2.3 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into LIMS

(when available) or other database so that accurate historical control limits can be generated.

9.2.4 Refer to the QC program document (QA-003) for further details of control limits.

9.3 Method Blank

9.3.1 A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001NC for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common lab contaminants, see below). Any blank contamination above the reporting limit must be less than 1/10 of the measured concentration of any sample in the associated preparation batch.

9.3.1.1 If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.

9.3.1.2 Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

9.3.1.3 If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client. NOTE: For Ohio VAP work, there can be no target analyte greater than the RL.

9.3.2 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.3.3 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.4 Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective actions.

9.4 Laboratory Control Sample (LCS)

9.4.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All control analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency.

9.4.2 If any control analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

9.4.2.1 If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).

9.4.2.2 If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.4.3 Ongoing monitoring of the LCS over time provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.4.4 Additionally, when an all-analyte check sample is used, all non-controlling compounds must attain a recovery of 5% or greater if the compound is on the client's list.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1 A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

9.5.1.1 If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

9.5.1.2 If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

9.5.1.3 The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.6 Surrogates

9.6.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.6.2 If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

9.6.2.1 Check all calculations for error.

9.6.2.2 Ensure that instrument performance is acceptable.

9.6.2.3 Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.

9.6.2.3.1 It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect. **Note:** If all associated QC meets criteria (blank, LCS/LCSD), up to one surrogate per fraction may be outside of acceptance criteria , as long as the recovery is greater than 10%. **Note:** For Ohio VAP and DoD samples, all surrogates must be within acceptance criteria.

9.6.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.6.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.6.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a minimum five-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

10.3.1 At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.2 Inject the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.3 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Each day Benzidine is to be determined, the tailing factor must be less than 3.0. The tailing factor for pentachlorophenol must be less than 5. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be < 20%. Refer to Section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. Quadratic fit may NOT be used for samples analyzed under South Carolina Certification. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 2 ng on column. (For example, 5 uL of 80ppm IS mix is added to 100 uL of extract. This results in 4 ng, but only 0.5ul is injected, resulting in a final on column amount of 2 ng.)The concentration ranges of all analytes are listed in Tables 12 and 13.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Section 12 and verify that the CCC and SPCC criteria in Sections 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.6 Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.6.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.6.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation.

10.4.7.1 If an analyte in the initial calibration is > 15%, then calibration on a curve must be used. Linear or quadratic curve fits may be used. The analyst should consider instrument maintenance to improve the linearity of response. Use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. If the % RSD is >15%, the analyst may drop the low or high points in the ICAL, as long as a minimum of 5 points are maintained and the quantitation range is adjusted accordingly. If the % RSD is still >15%, a quadratic or linear curve may be used. The coefficient of determination (r^2) must be ≥ 0.990. If the coefficient of determination is < 0.990, then any hits for these compounds must be flagged as estimated. If a curve is not linear for any compound that is found in a samples, the result must be flagged as estimated. Linear is defined as <15% RSD or a coefficient of determination of 0.990.

10.4.7.2 Note: Several components do not respond well by this method (poor linearity). These compounds are indene, acrylamide, 4-Nitroquinoline-1-oxide, famphur, benzenethiol, kepone, and 2,4-toluenediamine. If these compounds are requested by a client and hits are found, alternate standards or methods will be needed for more accurate quantitation. Sensitivity as demonstrated by the low standard is sufficient to substantiate a non-detect.

10.4.8 If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9 **Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.**

10.5 Initial Calibration Verification (ICV)

10.5.1 Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. The recovery CCC compounds must be ≤ 20%. The recovery for non-CCC compounds must be ≤ 50% with an allowance of up to 6 compounds >50%.

10.6 Continuing Calibration

10.6.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. The injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.6.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.6.3 The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05.
- The percent difference or drift of the CCC compounds from the initial calibration must be ≤ 20%. (see Section 12 for calculations) In addition, the percent difference or drift of all analytes must be ≤ 50%, with allowance for up to (4) compounds to be greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.
- NOTE: There is no internal standard criteria for samples. Criteria is only for continuing and initial calibrations.
- NOTE: Ohio VAP rules require that any sample with internal standard outliers be reanalyzed. The criteria for acceptance is between 50% and 200% of same internal standard in continuing calibration.

10.6.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.6.4. Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

11. PROCEDURE

11.1 Sample Preparation

11.1.1 Samples are prepared following SOP CORP-OP-0001NC.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in Section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 2 ng injected on column. Mix thoroughly before injection into the instrument.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. Chromatograms before and after manual integration are required by many programs.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in Section

11.3 Dilutions

11.3.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1 Guidance for Dilutions Due to Matrix

11.3.1.1 If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed

at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 Reporting Dilutions

11.3.2.1 The most concentrated dilution with target compounds within the calibration range will be reported. Other dilutions will only be reported at client request.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 $\pm$ 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5 Retention time criteria for samples

11.5.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.5.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6 Procedural Variations

11.6.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.7 Troubleshooting Guide

11.7.1 Daily Instrument Maintenance

11.7.1.1 In addition to the checks listed in the instrument maintenance schedule in the STL North Canton Laboratory Quality Manual (LQM), current version, the following daily maintenance should be performed.

11.7.1.1.1 Clip Column as necessary.

11.7.1.1.2 Install new or cleaned injection port liner as necessary.

11.7.1.1.3 Install new septum as necessary.

11.7.1.1.4 Perform autotune.

11.7.2 Major Maintenance

11.7.2.1 A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

12.1.1.1 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

12.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.1.1.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.1.4 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 Mass chromatogram searches.

12.2.1 Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1.1 Hexachlorophene

12.2.1.1.1 Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

12.3.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.3.2 The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)

12.3.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.3.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.

12.3.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.3.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.3.7 Note: For water samples, the TIC searches begin with compounds eluting after the first surrogate (2-Fluorophenol). For solid samples, the TIC searches begin with compounds eluting after the Aldol Condensation Product. Any compounds eluting before these analytes are considered volatile analytes are reported in the volatile analysis. A possible exception to this general rule would be if an early eluting compound was the reason for a sample dilution.

12.3.8 If a client requests 10 TICs, the laboratory supplies a minimum of 10. For a request of 20 TICS, the laboratory would supply a minimum of 20, assuming that number of compounds were available.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$RF =$ Mean of RFs from intial caibration for a compound

$SD =$ Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

$RFi =$ RF for each of the calibration levels

$N =$ Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

$C_{actual} =$ Known concentration in standard

$C_{found} =$ Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is ≤ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.5.3.2 Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where: C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}^2\right)$$

Where: C = Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where: V_t= Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are

combined, $V_t = 2{,}000$.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

Where: W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or one for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where: S_{SR} = Spike sample result

S_R=Sample result

S_A = Spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where: RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where: A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is} =Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x =Area of the total ion chromatogram for the compound being measured

A_{is}=Area of the total ion chromatogram for the nearest internal standard without interference

RF=1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

13. METHOD PERFORMANCE

13.1 Method Detection Limit

13.1.1 Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in policy S-Q-003 and SOP NC-QA-0021.

13.2 Initial Demonstration

13.2.1 Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.1.3 If any analyte does not meet the acceptance criteria the test must be repeated.

Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Non-standard analytes

13.3.1 For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

13.4 Training Qualification

13.4.1 The Group/Team Leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.4.2 Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15. WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.3 Waste Streams Produced by the Method

15.3.1 **Vials containing sample extracts:** These vials are placed in the vial waste located in the GC/MS laboratory.

16. REFERENCES

16.1 References

16.1.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.1.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

16.1.3 Corporate Quality Management Plan (QMP), current version.

16.1.4 STL Laboratory Quality Manual (LQM), current version.

16.2 Associated SOPs and Policies, latest version

16.2.1 QA Policy, QA-003

16.2.2 Glassware Washing, NC-QA-0014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5 Supplemental Practices for DoD Project Work, SOP, NC-QA-0016

16.2.6 Standard and Reagents, SOP NC-QA-0017.

17. MISCELLANEOUS

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions from compounds have been changed from those recommennded in SW-846 in order to improve the reliability of qualitative identification.

17.2 Tables

Table 1 - STL North Canton Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg	TCLP mg/L
1,1-Biphenyl	92-52-4	10	330	1	50	
1,2,4-Trichlorobenzene	120-82-1	10	330	1	50	
1,2-Dichlorobenzene	95-50-1	10	330	1	50	
1,3-Dichlorobenzene	541-73-1	10	330	1	50	
1,4-Dichlorobenzene	106-46-7	10	330	1	50	0.004
1-Methyl Naphthalene	90-12-0	10	330	0.2	6.67	
2,2'-oxybis(1-chloropropane) [1]	108-60-1	10	330	2	100	
2,4,5-Trichlorophenol	95-95-4	10	330	1	150	0.02
2,4,6-Trichlorophenol	88-06-2	10	330	1	150	0.02
2,4-Dichlorophenol	120-83-2	10	330	2	150	
2,4-Dimethylphenol	105-67-9	10	330	2	150	
2,4-Dinitrophenol	51-28-5	50	1600	5	330	
2,4-Dinitrotoluene	121-14-2	10	330	5	200	0.02
2,6-Dinitrotoluene	606-20-2	10	330	5	200	
2-Chloronaphthalene	91-58-7	10	330	1	50	
2-Chlorophenol	95-57-8	10	330	1	50	
2-Methylnaphthalene	91-57-6	10	330	0.2	6.67	
2-Methylphenol	95-48-7	10	330	1	200	
2-Nitroaniline	88-74-4	50	1600	2	200	
2-Nitrophenol	88-75-5	10	330	2	50	
3,3'-Dichlorobenzidine	91-94-1	50	1600	5	100	
3-Nitroaniline	99-09-2	50	1600	2	200	
4,6-Dinitro-2-methylphenol	534-52-1	50	1600	5	150	
4-Bromophenyl phenyl ether	101-55-3	10	330	2	50	
4-Chloro-3-methylphenol	59-50-7	10	330	2	150	
4-Chloroaniline	106-47-8	10	330	2	150	
4-Chlorophenyl phenyl ether	7005-72-3	10	330	2	50	
4-Methylphenol	106-44-5	10	330	1	200	
4-Nitroaniline	100-01-6	50	1600	2	200	
4-Nitrophenol	100-02-7	50	1600	5	330	
Acenaphthene	83-32-9	10	330	0.2	6.67	
Acenaphthylene	208-96-8	10	330	0.2	6.67	
Aniline	62-53-3	10	330	1	330	
Anthracene	120-12-7	10	330	0.2	6.67	
Atrazine	1912-24-9	10	330	1	200	
Azobenzene	103-33-3	10	330	10	330	
Benzaldehyde	100-52-7	10	330	1	100	
Benzenethiol	108-98-5	10	330	10	330	
Benzidine	92-87-5	100	3300	5	330	
Benzo(a)anthracene	56-55-3	10	330	0.2	6.67	
Benzo(a)pyrene	50-32-8	10	330	0.2	6.67	
Benzo(b)fluoranthene	205-99-2	10	330	0.2	6.67	
Benzo(g,h,i)perylene	191-24-2	10	330	0.2	6.67	
Benzo(k)fluoranthene	207-08-9	10	330	0.2	6.67	
Benzoic acid	65-85-0	50	1600	10	660	

Table 1 (Cont'd)

Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg	TCLP mg/L
Benzyl alcohol	100-51-6	10	330	5	330	
Bis(2-chloroethoxy)methane	111-91-1	10	330	1	100	
Bis(2-chloroethyl)ether	111-44-4	10	330	1	100	
Bis(2-ethylhexyl)phthalate	117-81-7	10	330	1	50	
Butyl benzyl phthalate	85-68-7	10	330	1	50	
Caprolactam	105-60-2	10	330	5	330	
Carbazole	86-74-8	10	330	1	50	
Chrysene	218-01-9	10	330	0.2	6.67	
Dibenz(a,h)anthracene	53-70-3	10	330	0.2	6.67	
Dibenzofuran	132-64-9	10	330	1	50	
Diethylphthalate	84-66-2	10	330	1	50	
Dimethyl phthalate	131-11-3	10	330	1	50	
Di-n-butyl phthalate	84-74-2	10	330	1	50	
Di-n-octylphthalate	117-84-0	10	330	1	50	
Fluoranthene	206-44-0	10	330	0.2	6.67	
Fluorene	86-73-7	10	330	0.2	6.67	
Hexachlorobenzene	118-74-1	10	330	0.2	6.67	0.02
Hexachlorobutadiene	87-68-3	10	330	1	50	0.02
Hexachlorocyclopentadiene	77-47-4	50	1600	10	330	0.02
Hexachloroethane	67-72-1	10	330	1	50	
Indene	95-13-6	10	330	5	330	
Indeno(1,2,3-cd)pyrene	193-39-5	10	330	0.2	6.67	
Isophorone	78-59-1	10	330	1	50	
Naphthalene	91-20-3	10	330	0.2	6.67	
Nitrobenzene	98-95-3	10	330	1	100	0.004
N-nitrosodimethylamine	62-75-9	10	330	1	100	
N-Nitroso-di-n-propylamine	621-64-7	10	330	1	50	
N-Nitrosodiphenylamine	86-30-6	10	330	1	50	
Pentachlorophenol	87-86-5	50	1600	5	150	0.04
Phenanthrene	85-01-8	10	330	0.2	6.67	
Phenol	108-95-2	10	330	1	50	
Pyrene	129-00-0	10	330	0.2	6.67	
Pyridine	110-86-1	20	660	1	100	0.02
Quinoline	91-22-5	10	330	5	330	
m-Cresol & p Cresol						0.04
o-Cresol						0.004

[1] 2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether.

Table 2 - STL North Canton Appendix IX[1] Standard Reporting Limits

Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	1	100
1,3,5-Trinitrobenzene	99-35-4	50	1600	5	1600
1,3-Dinitrobenzene	99-65-0	10	330	2	330
1,4-Dinitrobenzene	100-25-4	10	330	2	100
1,4-Naphthoquinone	130-15-4	50	1600	50	330
1-Naphthylamine	134-32-7	10	330	2	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600	10	100
2,6-Dichlorophenol	87-65-0	10	330	5	150
2-Acetylaminofluorene	53-96-3	100	3300	10	330
2-Naphthylamine	91-59-8	10	330	2	200
2-Picoline	109-06-8	20	660	5	330
2-secbutyl-4,6-dinitrophenol (Dinoseb2)	88-85-7	20	660	2	330
3,3'-Dimethylbenzidine	119-93-7	50	1600	5	330
3-Methylcholanthrene	56-49-5	20	660	5	200
3-Methylphenol	108-39-4	10	330	1	200
4-Aminobiphenyl	92-67-1	50	1600	5	330
4-Nitroquinoline-1-oxide	56-57-5	100	3300	5	330
5-Nitro-o-toluidine	99-55-8	20	660	2	330
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660	2	330
a,a-Dimethyl-phenethylamine	122-09-8	50	1600	5	660
Acetophenone	98-86-2	10	330	1	100
Aramite	140-57-8	20	660	5	330
Diallate[2]	2303-16-4	20	660	10	330
Dibenz(a,j)acridine	224-42-0	20	660	5	330
Dimethoate	60-51-5	20	660	2	330
Disulfoton	298-04-4	50	1600	2	330
Ethyl methanesulfonate	62-50-0	10	330	2	330
Famphur	52-85-7	100	3300	10	3300
Hexachloropropene	1888-71-7	100	3300	5	0.02
Isosafrole	120-58-1	20	660	5	330
Methapyrilene	91-80-5	50	1600	2	330
Methyl methanesulfonate	66-27-3	10	330	2	330
N-Nitrosodiethylamine	55-18-5	10	330	2	100
n-Nitrosodi-n-butylamine	924-16-3	10	330	2	100
N-Nitrosomethylethylamine	10595-95-6	10	330	2	100
N-Nitrosomorpholine	59-89-2	10	330	2	330
N-Nitrosopiperidine	100-75-4	10	330	2	330
N-Nitrosopyrrolidine	930-55-2	10	330	2	50
o,o,o-Triethyl-Phosphorothioate	126-68-1	50	1600	2	330
o-Toluidine	95-53-4	20	660	2	330
p-(Dimethylamino)azobenzene	60-11-7	20	660	2	330
p-Chlorobenzilate	510-15-6	10	330	2	330
Pentachlorobenzene	608-93-5	10	330	2	100

Table 2 (Cont'd)

Analytes	CAS Number	Water µg/L	Soil µg/kg	Low Level Water, µg/L	Low Level Soil, µg/kg
Pentachloroethane	76-01-7	50	1600	20	330
Pentachloronitrobenzene	82-68-8	50	1600	2	330
Phenacetin	62-44-2	20	660	2	330
Phorate	298-02-2	50	1600	2	330
p-Phenylenediamine	106-50-3	100	3300	40	660
Pronamide	23950-58-5	20	660	2	330
Safrole	94-59-7	20	660	2	330
Sulfotepp	3689-24-5	50	1600	5	330
Thionazin	297-97-2	50	1600	2	330
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	1	100
1,3,5-Trinitrobenzene	99-35-4	50	1600	5	1600
1,3-Dinitrobenzene	99-65-0	10	330	2	330
1,4-Dinitrobenzene	100-25-4	10	330	2	100

[1] The Appendix IX standard contains additional analytes required for the Appendix IX list. The STL North Canton primary standard must also be analyzed to include all of the Appendix IX list

Table 2A – STL North Canton Michigan Program[1]

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Acenaphthene	83-32-9	5	330
Acenaphthylene	208-96-8	5	330
Acetophenone	98-86-2	5	330
Anthracene	120-12-7	5	330
Atrzine	1912-24-9	5	330
Benzaldehyde	100-52-7	10	330
Benzo(a)anthracene	56-55-3	1	330
Benzo(a)pyrene	50-32-8	2	330
Benzo(b)fluoranthene	205-99-2	2	330
Benzo(g,h,i)perylene	191-24-2	5	330
Benzo(k)fluoranthene	207-08-9	5	330
1,1'-Biphenyl	92-52-4	10	330
4-Bromophenylphenyl ehter	101-55-3	5	330
Butylbenzylphthalate	85-68-7	5	330
di-n-Butylphthalate	84-74-2	5	330
Caprolactam	105-60-2	10	330
Carbazole	86-74-8	10	330
4-Chloroaniline	106-47-8	20	1700
bis(2-Chloroethoxy)methane	111-91-1	5	330
bis(2-Chloroethyl)ether	111-44-4	4	330
bis(2-Chloroisopropyl)ether	108-60-1	5	330
4-Chloro-3-Methylphenol	59-50-7	5	330
2-Chloronaphthalene	91-58-7	5	330
2-Chlorophenol	95-57-8	5	330
4-Chlorophenyl phenyl ether	7005-72-3	5	330
Chrysene	218-01-9	5	330
Dibenz(a,h)anthracene	53-70-3	2	330
Dibenzofuran	132-64-9	5	330
3,3'-Dichlorobenzidine	91-94-1	4	2000
2,4-Dichlorophenol	120-83-2	10	330
Diethylphthalate	84-66-2	5	330
2-4-Dimethylphenol	105-67-9	5	330
Dimethylphthalate	131-11-3	5	330
4,6-Dinitro-2-methylphenol	534-52-1	20	1700
2,4-Dinitrophenol	51-28-5	20	1700
2,4-Dinitrotoluene	121-14-2	5	330
2,6-Dinitrotoluene	606-20-2	5	330
bis(2-Ethylhexyl)phthalate	117-81-7	5	330
Fluoranthene	206-44-0	5	330
Fluorene	86-73-7	5	330
Hexachlorobenzene	118-74-1	5	330
Hexachlorobutadiene	87-68-3	5	330
Hexachlorocyclopentadiene	77-47-4	5	330
Hexachloroethane	67-72-1	5	330

Table 2A (Cont'd)

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Indeno(1,2,3-cd)pyrene	193-39-5	2	330
Isophorone	78-59-1	5	330
2-Methylnapthalene	91-57-6	5	330
2-Methylphenol	95-48-7	5	330
4-Methylphenol	106-44-5	5	330
Naphthalene	91-20-3	5	330
2-Nitroaniline	88-74-4	20	1700
3-Nitroaniline	99-09-2	20	1700
4-Nitroaniline	100-01-6	20	1700
Nitrobenzene	95-95-3	4	330
2-Nitrophenol	88-75-5	5	330
4-Nitrophenol	100-02-7	20	1700
N-Nitroso-di-n-propylamine	621-64-7	5	330
N-Nitrosodiphenylamine (diphenylamine)	62-75-9	5	330
di-n-Octylphthalate	117-84-0	5	330
Pentachlorophenol	87-86-5	20	800
Phenanthrene	85-01-8	5	330
Phenol	108-95-2	5	330
Pyrene	129-00-0	5	330
2,4,5-Trichlorophenol	95-95-4	5	330
2,4,6-Trichlorophenol	88-06-2	4	330

[1] Reporting Limits are only for samples performed under the Michigan program.

Table 3 - Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	TCLP	TCL	Appendix IX
Pyridine	110-86-1	X		X
N-nitrosodimethylamine	62-75-9			X
Aniline	62-53-3			X
Phenol	108-95-2		X	X
Bis(2-chloroethyl)ether	111-44-4		X	X
2-Chlorophenol	95-57-8		X	X
1,3-Dichlorobenzene	541-73-1		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X
Benzyl alcohol	100-51-6			X
1,2-Dichlorobenzene	95-50-1		X	X
2-Methylphenol	95-48-7	X	X	X
2,2'-oxybis(1-chloropropane)	180-60-1		X	X
4-Methylphenol	106-44-5	X	X	X
N-Nitroso-di-n-propylamine	621-64-7		X	X
Hexachloroethane	67-72-1	X	X	X
Nitrobenzene	98-95-3	X	X	X
Isophorone	78-59-1		X	X
2-Nitrophenol	88-75-5		X	X
2,4-Dimethylphenol	105-67-9		X	X
Benzoic acid	65-85-0			
Bis(2-chloroethoxy)methane	111-91-1		X	X
2,4-Dichlorophenol	120-83-2		X	X
1,2,4-Trichlorobenzene	120-82-1		X	X
Naphthalene	91-20-3		X	X
4-Chloroaniline	106-47-8		X	X
Hexachlorobutadiene	87-68-3	X	X	X
4-Chloro-3-methylphenol	59-50-7		X	X
2-Methylnaphthalene	91-57-6		X	X
Hexachlorocyclopentadiene	77-47-4		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X
2-Chloronaphthalene	91-58-7		X	X
2-Nitroaniline	88-74-4		X	X
Dimethyl phthalate	131-11-3		X	X
Acenaphthylene	208-96-8		X	X
3-Nitroaniline	99-09-2		X	X
Acenaphthene	83-32-9		X	X
2,4-Dinitrophenol	51-28-5		X	X
4-Nitrophenol	100-02-7		X	X
Dibenzofuran	132-64-9		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X

1 Azobenzene is formed by decomposition of 1,2-diphenyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 3 (Cont'd)

Analyte	CAS Number	TCLP	TCL	Appendix IX
2,6-Dinitrotoluene	606-20-2		X	X
Diethylphthalate	84-66-2		X	X
4-Chlorophenyl phenyl ether	7005-72-3		X	X
Fluorene	86-73-7		X	X
4-Nitroaniline	100-01-6		X	X
4,6-Dinitro-2-methylphenol	534-52-1		X	X
N-Nitrosodiphenylamine	86-30-6		X	X
Azobenzene[1]	103-33-3			
4-Bromophenyl phenyl ether	101-55-3		X	X
Hexachlorobenzene	118-74-1	X	X	X
Pentachlorophenol	87-86-5	X	X	X
Phenanthrene	85-01-8		X	X
Anthracene	120-12-7		X	X
Carbazole	86-74-8		X	
	84-74-2		X	X
Fluoranthene	206-44-0		X	X
Benzidine	92-87-5			
Pyrene	129-00-0		X	X
Butyl benzyl phthalate	85-68-7		X	X
3,3'-Dichlorobenzidine	91-94-1		X	X
Benzo(a)anthracene	56-55-3		X	X
Bis(2-ethylhexyl)phthalate	117-81-7		X	X
Chrysene	218-01-9		X	X
Di-n-octylphthalate	117-84-0		X	X
Benzo(b)fluoranthene	205-99-2		X	X
Benzo(k)fluoranthene	207-08-9		X	X
Benzo(a)pyrene	50-32-8		X	X
Indeno(1,2,3-cd)pyrene	193-39-5		X	X
Dibenz(a,h)anthracene	53-70-3		X	X
Benzo(g,h,i)perylene	191-24-2		X	X
Benzaldehyde	100-52-7		X	
Caprolactam	105-60-2		X	
1,1-Biphenyl	92-52-4		X	
Atrazine	1912-24-9		X	

[1] Azobenzene is formed by decomposition of 1,2-diphenyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4 - Reportable Analytes for STL North Canton Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	TCLP	TCL	Appendix IX
2-Picoline	109-06-8			X
N-Nitrosomethylethylamine	10595-95-6			X
Methyl methanesulfonate	66-27-3			X
N-Nitrosodiethylamine	55-18-5			X
Ethyl methanesulfonate	62-50-0			X
Pentachloroethane	76-01-7			X
Acetophenone	98-86-2		X	X
N-Nitrosopyrrolidine	930-55-2			X
N-Nitrosomorpholine	59-89-2			X
o-Toluidine	95-53-4			X
3-Methylphenol	108-39-4			X
N-Nitrosopiperidine	100-75-4			X
o,o,o-Triethyl-Phosphorothioate	126-68-1			X
a,a-Dimethyl-phenethylamine	122-09-8			X
2,6-Dichlorophenol	87-65-0			X
Hexachloropropene	1888-71-7			X
p-Phenylenediamine	106-50-3			X
n-Nitrosodi-n-butylamine	924-16-3			X
Safrole	94-59-7			X
1,2,4,5-Tetrachlorobenzene	95-94-3			X
Isosafrole	120-58-1			X
1,4-Dinitrobenzene	100-25-4			X
1,4-Naphthoquinone	130-15-4			X
1,3-Dinitrobenzene	99-65-0			X
Pentachlorobenzene	608-93-5			X
1-Naphthylamine	134-32-7			X
2-Naphthylamine	91-59-8			X
2,3,4,6-Tetrachlorophenol	58-90-2			X
5-Nitro-o-toluidine	99-55-8			X
Thionazin	297-97-2			X
1,3,5-Trinitrobenzene	99-35-4			X
Sulfotepp	3689-24-5			X
Phorate	298-02-2			X
Phenacetin	62-44-2			X
Diallate	2303-16-4			X
Dimethoate	60-51-5			X
4-Aminobiphenyl	92-67-1			X
Pentachloronitrobenzene	82-68-8			X
Pronamide	23950-58-5			X
Disulfoton	298-04-4			X
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7			X

Table 4 (Cont'd)

Semivolatiles	CAS Number	TCLP	TCL	Appendix IX
4-Nitroquinoline-1-oxide	56-57-5			X
Famphur	52-85-7			X
Methapyrilene	91-80-5			X
Aramite	140-57-8			X
p-(Dimethylamino)azobenzene	60-11-7			X
p-Chlorobenzilate	510-15-6			X
3,3'-Dimethylbenzidine	119-93-7			X
2-Acetylaminofluorene	53-96-3			X
Dibenz(a,j)acridine[1]	224-42-0			
7,12-Dimethylbenz(a)anthracene	57-97-6			X
3-Methylcholanthrene	56-49-5			X
Hexachlorophene[2]	70-30-4			X
Diphenylamine[3]	122-39-4			X

[1] Skinner List Compound

[2] Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See Section 12.2.1)

[3] Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore, these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Table 5 - Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	45°C for 1 minutes
Column Temperature Program	45- 100°C at 25°C/min for 0 min 100 - 280°C at 30°C/min for 0 min 280 - 100°C at 25°C/min for 2 min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's Specifications
Injector	Grob-type, split / splitless
Sample Volume	0.5 µl
Carrier Gas	Helium at 30 cm/sec

Table 6 - DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 – 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 – 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 – 9% of mass 198
275	10 – 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 – 23% of mass 442

Table 7 - Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Benzaldehyde	77	105	106
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
Caprolactam	113	55	56
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
1,1'-Biphenyl	154	153	76

Table 7 (Cont'd)

Analyte	Primary	Secondary	Tertiary
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Atrazine	200	173	215
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125

Table 7 (Cont'd)

Analyte	Primary	Secondary	Tertiary
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8 - Additional Appendix IX Analytes in Approximate Retention Time Order and Characteristic Ions

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147

Table 8 (Cont'd)

Analyte	Primary	Secondary	Tertiary
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9 - 8270C LCS Control Compounds

LCS Compounds	Spiking Level, Conc. Added = 20 ug/L
1,2,4-Trichlorobenzene	20
Acenaphthene	20
2,4-Dinitrotoluene	20
Pyrene	20
N-Nitroso-di-n-propylamine	20
1,4-Dichlorobenzene	20
Pentachlorophenol	20
Phenol	20
2-Chlorophenol	20
4-Chloro-3-methylphenol	20
4-Nitrophenol	20

Table 9A 8270C All Analyte Spike Mix	
Acenaphthene	100
Acenaphthylene	100
Anthracene	100
Benzo(a)anthracene	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Benzo(ghi)perylene	100
Benzyl butyl phthalate	100
Bis(2-chloroethyl)ether	100
Bis(2-chloroethoxy)methane	100
Bis(2-ethylhexyl)phthalate	100
Bis(2-chloroisopropyl)ether	100
4-Bromophenyl phenyl ether	100
2-Chloronaphthalene	100
4-Chlorophenyl phenyl ether	100
Chrysene	100
Dibenzo(a,h)anthracene	100
Di-n-butylphthalate	100
1,3-Dichlorobenzene	100
1,2-Dichlorobenzene	100

Table 9A (Cont'd)

Table 9A 8270C All Analyte Spike Mix	
1,4-Dichlorobenzene	100
3,3'-Dichlorobenzidine	100
Diethyl phthalate	100
Dimethyl phthalate	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Di-n-octylphthalate	100
Fluoranthene	100
Fluorene	100
Hexachlorobenzene	100
Hexachlorobutadiene	100
Hexachloroethane	100
Indeno(1,2,3-cd)pyrene	100
Isophorone	100
Naphthalene	100
Nitrobenzene	100
N-Nitrosodi-n-propylamine	100
Phenanthrene	100
Pyrene	100
1,2,4-Trichlorobenzene	100
4-Chloro-3-methylphenol	100
2-Chlorophenol	100

Table 9A (Cont'd)

Table 9A 8270C All Analyte Spike Mix	
2,4-Dichlorophenol	100
2,4-Dimethylphenol	100
2,4-Dinitrophenol	100
2-Methyl-4,6-dinitrophenol	100
2-Nitrophenol	100
4-Nitrophenol	100
Pentachlorophenol	100
Phenol	100
2,4,6-Trichlorophenol	100
	100
Acetophenone	100
Atrazine	100
Caprolactam	100
Benzaldehyde	100
1,1′-Biphenyl	100
Benzoic Acid	100
1,4-Dioxane	100
Benzyl Alcohol	100
Carbazole	100
4-Chloroaniline	100
Dibenzofuran	100
Hexachlorocyclopentadiene	100
2-Methylnaphthalene	100
Quinoline	100
1-Methylnaphthalene	100

Table 9A 8270C All Analyte Spike Mix	
2-Methylphenol	100
4-Methylphenol	100
4-Nitroaniline	100
2-Nitroaniline	100
3-Nitroaniline	100
Pyridine	100
2,3,5,6-Tetrachlorophenol	100
2,4,5-Trichlorophenol	100
N-Nitrosodimethylamine	100
N-Nitrosodiphenylamine	100

Table 10 - TCLP LCS Compounds

LCS Compounds	Spiking Level, mg/L in extract
1,4-Dichlorobenzene	0.08
2,4-Dinitrotoluene	0.08
Hexachlorobenzene	0.08
Hexachlorobutadiene	0.08
Hexachloroethane	0.08
2-Methylphenol	0.08
3-Methylphenol	0.08
4-Methylphenol	0.08
Nitrobenzene	0.08
Pentachlorophenol	0.08
Pyridine	0.08
2,4,5-Trichlorophenol	0.08
2,4,6-Trichlorophenol	0.08

Recovery limits for the LCS and for matrix spikes are generated historical data, and are maintained by the QA Dept.

Table 11 - 8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, Conc. Added = 20 ug/L / 30 ug/L
Nitrobenzene-d5	20
2-Fluorobiphenyl	20
Terphenyl-d14	20
1,2-Dichlorobenzene-d4[1]	20
Phenol-d5	30
2-Fluorophenol	30
2,4,6-Tribromophenol	30
2-Chlorophenol-d4[1]	30

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12 - Calibration Ranges, µg/mL

Analyte	Calibration Range
Pyridine	0.25-12.5 ug/mL
N-nitrosodimethylamine	0.25-12.5 ug/mL
Aniline	0.25-12.5 ug/mL
Phenol	0.25-12.5 ug/mL
Bis(2-chloroethyl)ether	0.25-12.5 ug/mL
2-Chlorophenol	0.25-12.5 ug/mL
1,3-Dichlorobenzene	0.25-12.5 ug/mL
1,4-Dichlorobenzene	0.25-12.5 ug/mL
Benzyl alcohol	0.25-12.5 ug/mL
1,2-Dichlorobenzene	0.25-12.5 ug/mL
2-Methylphenol	0.25-12.5 ug/mL
2,2'-oxybis(1-chloropropane)[1]	0.25-12.5 ug/mL
4-Methylphenol	0.25-12.5 ug/mL
N-Nitroso-di-n-propylamine	0.25-12.5 ug/mL
Hexachloroethane	0.25-12.5 ug/mL
Nitrobenzene	0.25-12.5 ug/mL
Isophorone	0.25-12.5 ug/mL
2-Nitrophenol	0.25-12.5 ug/mL
2,4-Dimethylphenol	0.25-12.5 ug/mL
Benzoic acid	0.25-12.5 ug/mL
Bis(2-chloroethoxy)methane	0.25-12.5 ug/mL
2,4-Dichlorophenol	0.25-12.5 ug/mL
1,2,4-Trichlorobenzene	0.25-12.5 ug/mL
Naphthalene	0.05-10 ug/mL
4-Chloroaniline	0.25-12.5 ug/mL
Hexachlorobutadiene	0.25-12.5 ug/mL
4-Chloro-3-methylphenol	0.25-12.5 ug/mL
2-Methylnaphthalene	0.05-10 ug/mL
Hexachlorocyclopentadiene	0.25-12.5 ug/mL
2,4,6-Trichlorophenol	0.25-12.5 ug/mL
2,4,5-Trichlorophenol	0.25-12.5 ug/mL
2-Chloronaphthalene	0.25-12.5 ug/mL
2-Nitroaniline	0.25-12.5 ug/mL
Dimethyl phthalate	0.25-12.5 ug/mL
Acenaphthylene	0.05-10 ug/mL
3-Nitroaniline	0.25-12.5 ug/mL
Acenaphthene	0.05-10 ug/mL
2,4-Dinitrophenol	0.25-12.5 ug/mL
4-Nitrophenol	0.25-12.5 ug/mL
Dibenzofuran	0.25-12.5 ug/mL
2,4-Dinitrotoluene	0.25-12.5 ug/mL
2,6-Dinitrotoluene	0.25-12.5 ug/mL

Table 12 (Cont'd)

Analyte	Calibration Range
Diethylphthalate	0.25-12.5 ug/mL
4-Chlorophenyl phenyl ether	0.25-12.5 ug/mL
Fluorene	0.05-10 ug/mL
4-Nitroaniline	0.25-12.5 ug/mL
4,6-Dinitro-2-methylphenol	0.25-12.5 ug/mL
N-Nitrosodiphenylamine	0.25-12.5 ug/mL
Azobenzene[2]	0.25-12.5 ug/mL
4-Bromophenyl phenyl ether	0.25-12.5 ug/mL
Hexachlorobenzene	0.25-12.5 ug/mL
Pentachlorophenol	0.25-12.5 ug/mL
Phenanthrene	0.05-10 ug/mL
Anthracene	0.05-10 ug/mL
Carbazole	0.05-10 ug/mL
Di-n-butyl phthalate	0.25-12.5 ug/mL
Fluoranthene	0.05-10 ug/mL
Benzidine	0.25-12.5 ug/mL
Pyrene	0.05-10 ug/mL
Butyl benzyl phthalate	0.25-12.5 ug/mL
3,3'-Dichlorobenzidine	0.25-12.5 ug/mL
Benzo(a)anthracene	0.05-10 ug/mL
Bis(2-ethylhexyl)phthalate	0.25-12.5 ug/mL
Chrysene	0.05-10 ug/mL
Di-n-octylphthalate	0.25-12.5 ug/mL
Benzo(b)fluoranthene	0.05-10 ug/mL
Benzo(k)fluoranthene	0.05-10 ug/mL
Benzo(a)pyrene	0.05-10 ug/mL
Indeno(1,2,3-cd)pyrene	0.05-10 ug/mL
Dibenz(a,h)anthracene	0.05-10 ug/mL
Benzo(g,h,i)perylene	0.05-10 ug/mL
Benzaldehyde	0.25-12.5 ug/mL
Caprolactam	0.25-12.5 ug/mL
1,1'-Biphenyl	0.25-12.5 ug/mL
Atrazine	0.25-12.5 ug/mL

[1] 2,2'oxybis(1-chloropropane) was formerly known as bis(2-chloroisopropyl)ether

[2] Azobenzene is formed by decomposition of 1,2-diphenyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Note: Nine calibrations standards are prepared varying in concentration from 0.05 ug/mL to 12.5 ug/mL. A minimum of 5 calibration concentrations will be used for initial calibration . The concentration range of each analyte is listed in the table.

Table 13 - Calibration Ranges, Appendix IX, µg/mL

Semivolatiles	Calibration Range
2-Picoline	0.25-12.5 ug/mL
N-Nitrosomethylethylamine	0.25-12.5 ug/mL
Methyl methanesulfonate	0.25-12.5 ug/mL
N-Nitrosodiethylamine	0.25-12.5 ug/mL
Ethyl methanesulfonate	0.25-12.5 ug/mL
Pentachloroethane	0.25-12.5 ug/mL
Acetophenone	0.25-12.5 ug/mL
N-Nitrosopyrrolidine	0.25-12.5 ug/mL
N-Nitrosomorpholine	0.25-12.5 ug/mL
o-Toluidine	0.25-12.5 ug/mL
3-Methylphenol	0.25-12.5 ug/mL
N-Nitrosopiperidine	0.25-12.5 ug/mL
o,o,o-Triethyl-Phosphorothioate	0.25-12.5 ug/mL
a,a-Dimethyl-phenethylamine	0.25-12.5 ug/mL
2,6-Dichlorophenol	0.25-12.5 ug/mL
Hexachloropropene	0.25-12.5 ug/mL
p-Phenylenediamine	0.25-12.5 ug/mL
n-Nitrosodi-n-butylamine	0.25-12.5 ug/mL
Safrole	0.25-12.5 ug/mL
1,2,4,5-Tetrachlorobenzene	0.25-12.5 ug/mL
Isosafrole 1 + 2	0.25-12.5 ug/mL
1,4-Dinitrobenzene	0.25-12.5 ug/mL
1,4-Naphthoquinone	0.25-12.5 ug/mL
1,3-Dinitrobenzene	0.25-12.5 ug/mL
Pentachlorobenzene	0.25-12.5 ug/mL
1-Naphthylamine	0.25-12.5 ug/mL
2-Naphthylamine	0.25-12.5 ug/mL
2,3,4,6-Tetrachlorophenol	0.25-12.5 ug/mL
5-Nitro-o-toluidine	0.25-12.5 ug/mL
Thionazin	0.25-12.5 ug/mL
1,3,5-Trinitrobenzene	0.25-12.5 ug/mL
Sulfotepp	0.25-12.5 ug/mL
Phorate	0.25-12.5 ug/mL
Phenacetin	0.25-12.5 ug/mL
Diallate 1 + 2	0.25-12.5 ug/mL
Dimethoate	0.25-12.5 ug/mL
4-Aminobiphenyl	0.25-12.5 ug/mL
Pentachloronitrobenzene	0.25-12.5 ug/mL
Pronamide	0.25-12.5 ug/mL
Disulfoton	0.25-12.5 ug/mL
2-secbutyl-4,6-dinitrophenol (Dinoseb)	0.25-12.5 ug/mL
Methyl parathion	0.25-12.5 ug/mL
4-Nitroquinoline-1-oxide	0.25-12.5 ug/mL

Table 13 (Cont'd)

Semivolatiles	Calibration Range
Parathion	0.25-12.5 ug/mL
Isodrin	0.25-12.5 ug/mL
Kepone	0.25-12.5 ug/mL
Famphur	0.25-12.5 ug/mL
Methapyrilene	0.25-12.5 ug/mL
Aramite 1 and 2	0.25-12.5 ug/mL
p-(Dimethylamino)azobenzene	0.25-12.5 ug/mL
p-Chlorobenzilate	0.25-12.5 ug/mL
3,3'-Dimethylbenzidine	0.25-12.5 ug/mL
2-Acetylaminofluorene	0.25-12.5 ug/mL
Dibenz (a,j)acridine	0.25-12.5 ug/mL
7,12-Dimethylbenz(a)anthracene	0.25-12.5 ug/mL
3-Methylcholanthrene	0.25-12.5 ug/mL

Note: Nine calibrations standards are prepared varying in concentration from 0.05 ug/mL to 12.5 ug/mL. A minimum of 5 calibration concentrations will be used for initial calibration . The concentration range of each analyte is listed in the table.

Title: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHODS 8000B, 8021B, 8081A, 8082, 8151A, 8015B, AND 615

Approvals (Signature/Date):

Technology Specialist	Date 1/18/08	Health & Safety Coordinator	Date 1-18-08
Quality Assurance Manager	Date 1/18/08	Laboratory Director	Date 1/17/08
Technical Director	Date 1/18/08		

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1. SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices. Appendix E describes procedures for the analysis of petroleum hydrocarbons by SW-846 8015B methodology

2. SUMMARY OF METHOD

In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction or solid phase extraction (SOP # CORP-OP-0001NC). Solid samples are prepared using sonication, Soxhlet or automated Soxhlet (PCB only) (SOP # CORP-OP-0001NC). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the TestAmerica North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See the appendices for interferences specific to individual tests and suggested corrective actions.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. Eye protection that prevents splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Refer to the TestAmerica North Canton Corporate Safety Manual for a complete description of personal protection equipment. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. Latex, Nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.3. 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 (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.
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.
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.			

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable;** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. Opened containers of neat standards will be handled in a fume hood.

5.6. Sample extracts and standards, which are in a flammable solvent, shall be stored in an explosion-proof refrigerator.

5.7. When using hydrogen gas as a carrier, all precautions listed in the CSM shall be observed.

5.8. Standard preparation and dilution shall be performed inside an operating fume hood.

5.9. The gas chromatograph 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.

5.10. There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.11. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica assocaite. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

7.1.1. Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

7.1.2. Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at least every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration is an acceptable demonstration). Other volatile stock standards must be replaced every six months or sooner if comparison with check standards prepared from an independent source indicates a problem.

7.1.3. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If vendor-supplied standard has an earlier expiration date then that date is used. Refer to SOP NC-QA-0017, Standards and Reagents, for additional information.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

7.2.1.1. The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

7.2.2.1. Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at $\leq$6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Zero Air.

7.4. Quality control (QC) Standards

7.4.1. QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

8.1 Semivolatile extracts must be refrigerated at ≤6°C and analyzed within 40 days of the end of the extraction. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the batch definition.

9.2.1. Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD may be substituted.

9.3. Control Limits

In-house historical control limits may be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Project or program specific control limits may be used in place of in-house limits. Refer to policy QA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.4. Surrogates

All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. Surrogate recoveries must be met in the method blank (MB) and Laboratory Check Samples (LCS or LCS/LCSD). If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

  Note: For DoD QSM and Ohio VAP Projects, all surrogates must meet criteria.

9.4.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in-control result is reported.

9.4.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required. Re-preparation includes the parent sample and MS/MSD.

9.4.3. Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the corrective actions.

9.5. Method Blanks

For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatile samples, and sodium sulfate for semivolatile soils tests (Refer to SOP CORP-OP-0001NC for details). For low level volatiles, the method blank consists of reagent water. For medium level volatiles, the method blank consists of methanol as described in Appendix A. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.5.1. Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the corrective actions.

9.5.2. Refer to SOP NC-QA-0016 for further details concerning DoD Project Work.

9.6. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes with a subset of control analytes. If any control analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

9.6.1. Refer to the TestAmerica North Canton QC Program document (QA-003) for further details of the

corrective action.

9.6.2. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.6.3. LCS compound lists are included in the appendices.

9.7. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. The recovery for each spike of the pair must be within established control limits. If the RPD is out of control but both accuracy recoveries are within acceptance criteria, prepare an NCM, and qualify report.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.7.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

10. CALIBRATION AND STANDARDIZATION

Internal or external calibration may be used. Internal calibration is recommended unless the sample matrix is likely to interfere with the quantitation of the internal standard. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or FID jets or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.2. With the exception of Section 10.3 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least five points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

10.3. A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial or no purge run. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a

point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.4. External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$Calibration\ Factor\,(CF) = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected\,(ng)}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.5. Internal standard calibration

10.5.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If the sample matrix interferes with quantitation of the internal standard, then the external standard approach must be used instead. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.5.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard
C_s = Concentration of the analyte to be determined in the standard

10.6. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is $\leq$ 20%. The average %RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes. NOTE: This is

note allowed for Ohio VAP Projects.

10.6.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.6.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is $\leq$ 20%.

The equation for average response factor is:

$$Average\ response\ factor = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.6.3. Linear regression

The linear fit uses the following functions:

10.6.3.1. External Standard

$$y = ax + b$$

$$or$$

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.6.3.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_s}{A_{is}} - b\right]}{a}$$

Where: C_s = Concentration in the sample
A_s = Area of target peak in the sample
A_{is} = Area of internal standard in the sample
C_{is} = Concentration of the internal standard

10.6.4. Quadratic curve

The quadratic curve uses the following functions:

10.6.4.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.6.4.2. Internal Standard

$$y = a\left(\frac{A_s \times C_{is}}{A_{is}}\right) + c\left(\frac{A_s \times C_{is}}{A_{is}}\right)^2 + b$$

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard error (%RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows:

$$\% RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_i = True concentration for level i
PC_i = Predicted concentration for level i

Note that when average response factors are used, %RSE is equivalent to %RSD.

10.8. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the calculated intercept of the curve at zero response must be less than ± the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result a curve may have a very good correlation coefficient (>0.995), while also having > 100% error at the low point.

10.9. Weighting of data points

10.9.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.10. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

10.11. Calibration Verification

10.11.1. 12-hour Calibration

10.11.1.1. The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The center of each retention time window is updated with each 12-hour calibration or calibration verification.
NOTE: This is not acceptable for Ohio VAP Projects.

10.11.2. Calibration Verification

10.11.2.1. It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12-hour calibration with the exception that retention times are not updated.

10.11.3. Any individual compounds with %D < 15% meet the calibration criteria. The calibration verification is also acceptable if the average of the %D for all the analytes is < 15%. This average is calculated by summing the entire absolute %D results in the calibration (including

surrogates) and dividing by the number of analytes. Any analyte that is reportable as found must have a % difference of < 15% in the calibration verification or 12 hour calibration, on the column used for quantitation. Refer to section 12.1.2 for which result to report.

10.11.4. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.11.5. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. The bracketing standards on the column used for calibration must meet the same criteria as the opening standards. Bracketing is not necessary for internal standard methods.

10.11.6. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV then the proceeding samples have not been successfully bracketed but analysis may continue.

10.11.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed then the first will serve as the bracketing standard for the preceding samples and the last will serve as the CCV for the following samples.

10.11.8. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

10.11.9. Percent difference calculation

10.11.9.1. Percent difference for internal and external methods is calculated as follows:

Internal Standard

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where: RF_c and CF_c are the response and calibration factors from the continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response & calibration factors from the initial calibration

10.11.10. Percent drift calculation

10.11.10.1. Percent drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for percent drift are the same as for percent difference

$$\%\ Drift = \frac{Calculated\ Conc.\ -\ Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.11.11. Corrective Actions for Continuing Calibration

10.11.11.1. If the overall average percent drift of all analytes is greater than ± 15% corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average percent drift still varies by more than ± 15%, a new calibration curve must be prepared.

10.11.12. Corrective Action for Samples

10.11.12.1. For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be re-injected.

10.11.12.2. For external standard methods, any samples injected after the last good continuing calibration standard must be re-injected.

10.11.12.3. If the average percent drift for all the analytes in the calibration is over 15%; but all of the analytes requested for a particular sample have percent drift ≤ 15%, then the analysis is acceptable for that sample.

11. PROCEDURE

11.1. Extraction

11.1.1. Extraction procedures are referenced in the SOP CORP-OP-0001NC, latest revision.

11.2. Cleanup

11.2.1. Cleanup procedures are referenced in the SOP NC-OP-0025, latest revision.

11.3. Gas Chromatography

11.3.1. Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

11.4.1. In general, volatiles analytes are introduced using purge and trap as described in Appendix A.

11.4.2. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

11.5.1. An analytical sequence starts with an initial calibration or a calibration verification. Refer to the individual method appendices for method specific details of calibration verifications and analytical sequences.

11.5.2. The calibration verification includes analysis of standards containing all single response analytes and updating the retention time windows.

11.5.3. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a calibration verification.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three

retention times for each analyte (relative retention times may also be used). For multi-response analytes (e.g., Aroclors) use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid- point of the initial calibration and each 12-hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than +/- 0.05 minutes, use a retention time window appropriate for the analysis and run time. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Retention time studies are filed in the laboratory.

11.6.6. Corrective Action for Retention Times

11.6.6.1. The retention times of all compounds in the 12 hour calibration or calibration verification standard must be within the retention time window. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window.

11.6.6.2. The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

11.6.6.3. No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

11.7.1. The center of the retention time windows determined in Section 11.6 are adjusted to the retention time of each analyte as determined in the 12 hour calibration standards or continuing calibration verification standards. (See the method 8081A and 8082 appendices for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration or continuing calibration verification.

11.8. Procedural Variations

11.8.1. Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC Manager. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column, but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and/or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if J flag confirmation required).

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst:

A) The primary column approach, or
B) The better result approach

Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach:

12.1.2.2. The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is > 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for reanalysis.)

12.1.2.3. Better result approach

The higher of the two results is normally reported. The higher result is considered better because the higher result is generally higher because of chromatographic interference. The lower result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the higher result
- The continuing or bracketing calibration on the column with the higher result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for reanalysis.)

12.1.3. If the Relative Percent Difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{|R - R_2|}{\frac{1}{2}(R_1 + R_2)}$$

Where R=Result

12.1.4. Multi-response Analytes

For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

12.2.1. If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

12.3.1. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are detected, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions may be reported at client request if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgement and client site history will factors in the reporting of dual dilutions.

12.4. Interferences

12.4.1. If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria for Continuing Calibration

12.5.1. If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.6. Calculations

12.6.1. Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.2. External Standard Calculations

12.6.2.1. Aqueous samples

$$Concentration\ (mg / L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng, Section 10.1

12.6.2.2. Non-aqueous Samples

$$Concentration\ (mg / kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%\,Moisture}{100}$$ (D = 1 if wet weight is required)

12.6.3. Internal Standard Calculations

12.6.3.1. Aqueous Samples

$$Concentration\ (mg / L) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng
A_{is} = Response of the internal standard
RF = Response factor for analyte

12.6.3.2. Non-aqueous Samples

$$Concentration\ (mg / kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.4. Surrogate Recovery

12.6.4.1. Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.1.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOPs NC-QA-0021 and S-Q-003.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

13.3.1. The Group/Team Leader has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where

reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method.

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

15.2.1.1. **Vials containing sample extracts:** These vials are placed in the vial waste located in the GC/MS laboratory.

15.2.1.2. **Tubes containing sample extracts for TPH, Pesticides, PCBs and Herbicides**: These capped tubes are placed in the PCB/flammable wste located the GC prep laboratory.

15.2.1.3. **Samples, standards, and all extraction materials contaminated with high levels (>50ppm) of PCB's must be segregated into their own waste stream.** PCB wastes are collected in one of three waste streams, solid PCB, liquid PCB and PCB vial waste. PCB containing samples are located through a LIMS query and disposed of as PCB containing.

15.2.1.4. **Extracted solid samples contaminated with methylene chloride/acetone or acetone/hexane.** These materials are disposed of in the solid waste and debris in a red container located in the extractions lab.

15.2.1.5. **Discarded samples**. These samples are collected in the solid debris drum.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and Section 8000B

16.2 TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.3 TestAmerica Corporate Safety Manual, M-E-0001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.4 Associated SOPs and Policies, latest version

16.4.1 QA Policy, QA-003

16.4.2 Glassware Washing, NC-QA-0014

16.4.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.4.4 Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and NC-QA-0021

16.4.5 Standards and Reagents, NC-QA-0017

16.4.6 Supplemental Practices for DoD Project Work, NC-QA-0016

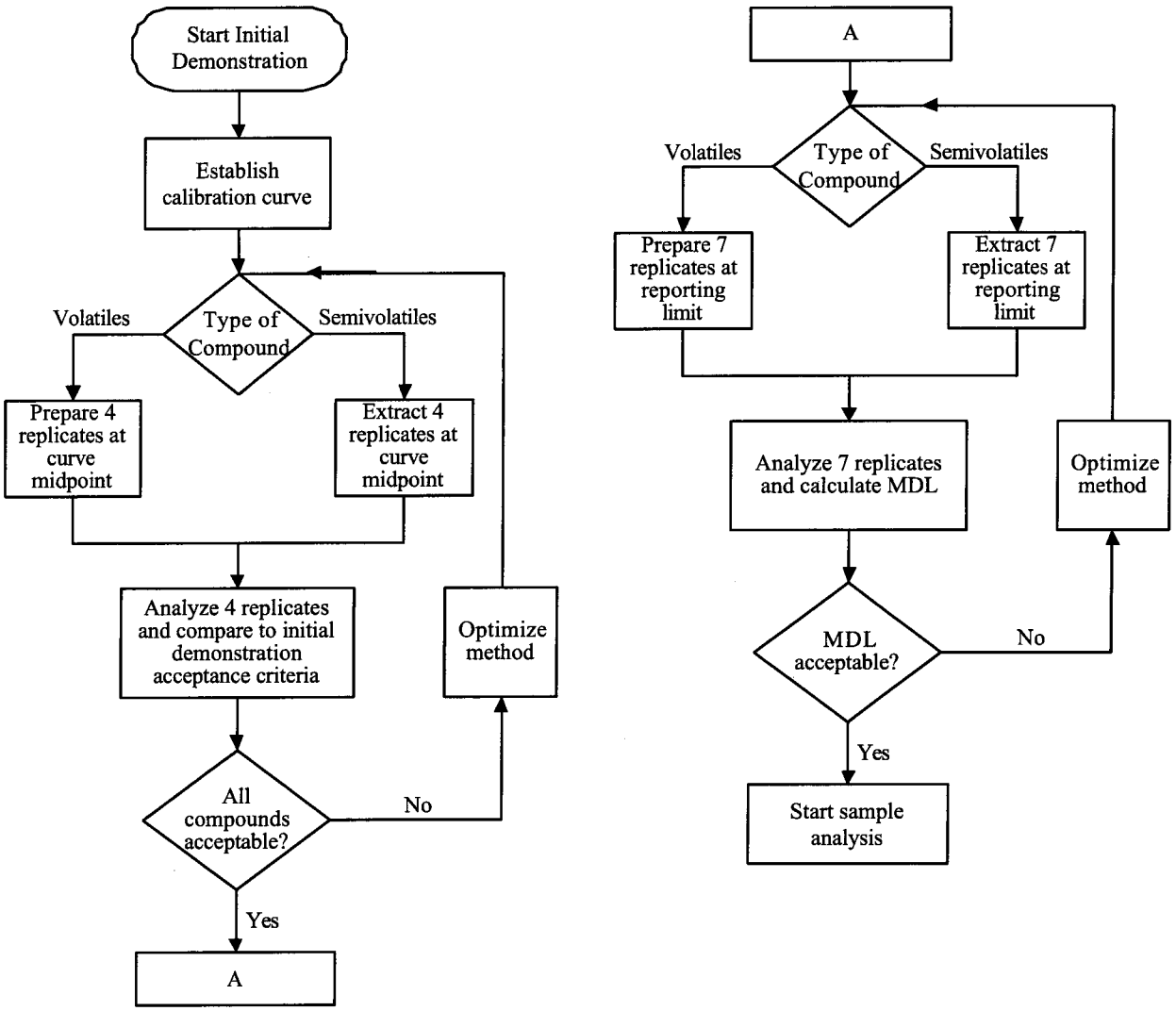
17. MISCELLANEOUS

17.1. Modifications from Reference Method

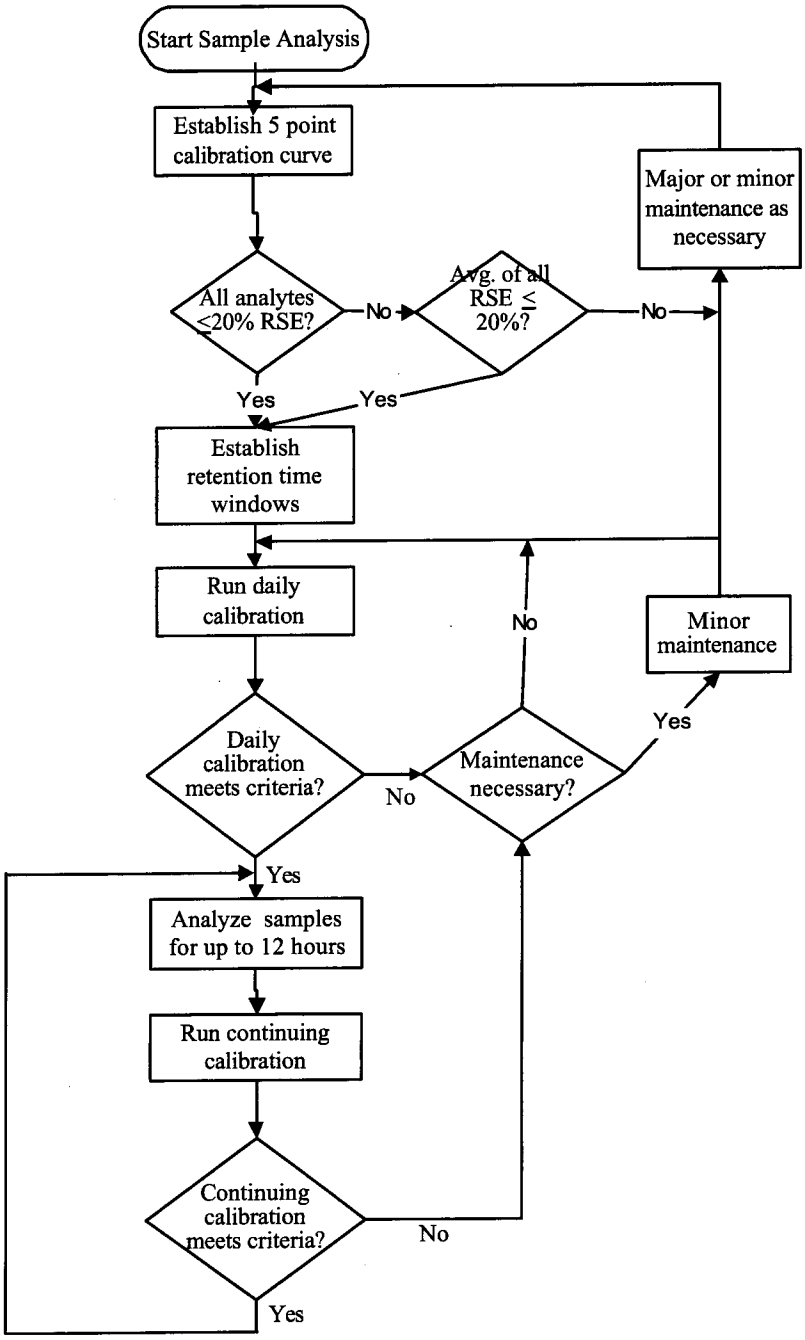
17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Flow Diagrams

17.2.1. Initial demonstration and MDL



17.2.2 Sample Analysis [1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

1.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure following Method 8021B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A-1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 200°C and are insoluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.

1.5. The associated LIMS method code is QR.

2. SUMMARY OF METHOD

2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low-level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and back-flushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photoionization Detector, an electrolytic conductivity detector or a combination of both.

2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge-and-trap GC following the normal water method. If very low detection limits are needed for soil samples then direct purge using sodium bisulfate preservation may be necessary.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this SOP.

4. INTERFERENCES

4.1. Refer to Section 4 of the method 8000B part of this SOP for general information on chromatographic interferences.

4.2. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank

prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.

4.5. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every 7 days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the 7-day period prior to the analysis of the contaminated holding blank is required.

4.6. Acidification of samples may result in hydrolysis of 2-chloroethyl-vinyl ether.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B section of this SOP for general safety requirements.

5.2. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: Benzene, Carbon Tetrachloride, 1,4-Dichlorobenzene, 1,2-Dichloroethane, Hexachlorobutadiene, 1,1,2,2-Tetrachloroethane, 1,1,2-Trichloroethane, Chloroform, 1,2-Dibromoethane, Tetrachloroethene, Trichloroethene, Vinyl Chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood.

5.3. GC VOA instruments use an ultraviolet (UV) light source, which must be shielded from view.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes -- 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These should be equipped with a 20 gauge (0.006" ID) needle. These will be used to measure and dispense methanolic solutions and aqueous samples.

6.2. Gas tight syringes -- 5 mL and 25 mL. Used for measuring sample volumes.

6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.

6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.

6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A-2 for suggested traps for specific tests.

6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.

6.7. Columns - Refer to Table A-2 for details of columns.

6.8. Volumetric flasks, Class A: 5 mL to 250 mL

6.9. pH paper – Range 0-14.

6.10. Balance capable of weighing to 0.01g for samples.

6.11. Chlorine Test Strips

6.12. Hach Chlorine Test Pillows

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Organic Free Water

7.2.1. Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. The suggested method for generating organic free water is continuously sparging water with helium or nitrogen.

7.2.2. Other methods may be used, so long as the requirement that the water show no interference is met. The procedure used should be documented in a lab specific attachment.

7.3. Methanol -- Purge and Trap Grade

7.4. Standards

7.4.1. Refer to Tables A-5 and A-6 for details of surrogate, matrix spiking and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low level standard must be equivalent to the reporting limits specified in Table A-1.

7.4.2. Volatile standards are prepared by injecting a measured volume of the stock standard into a syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. Unpreserved samples will be analyzed within seven days from sample collection.

8.3. Solid samples are field preserved with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared

VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle and store in a clean Teflon capped vial at 4+2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a VOA vial by adding 5 mL purge and trap grade methanol. (If a 25 g sample is to be used, add 25 mL methanol to the VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

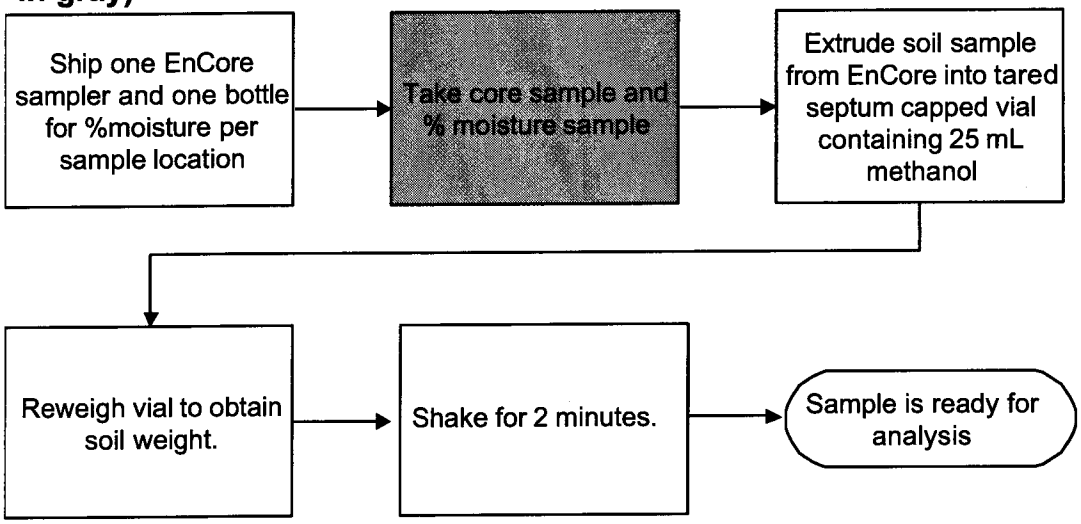
8.8. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at

4°C +/- 2°C. The extracts are stored with minimum headspace.

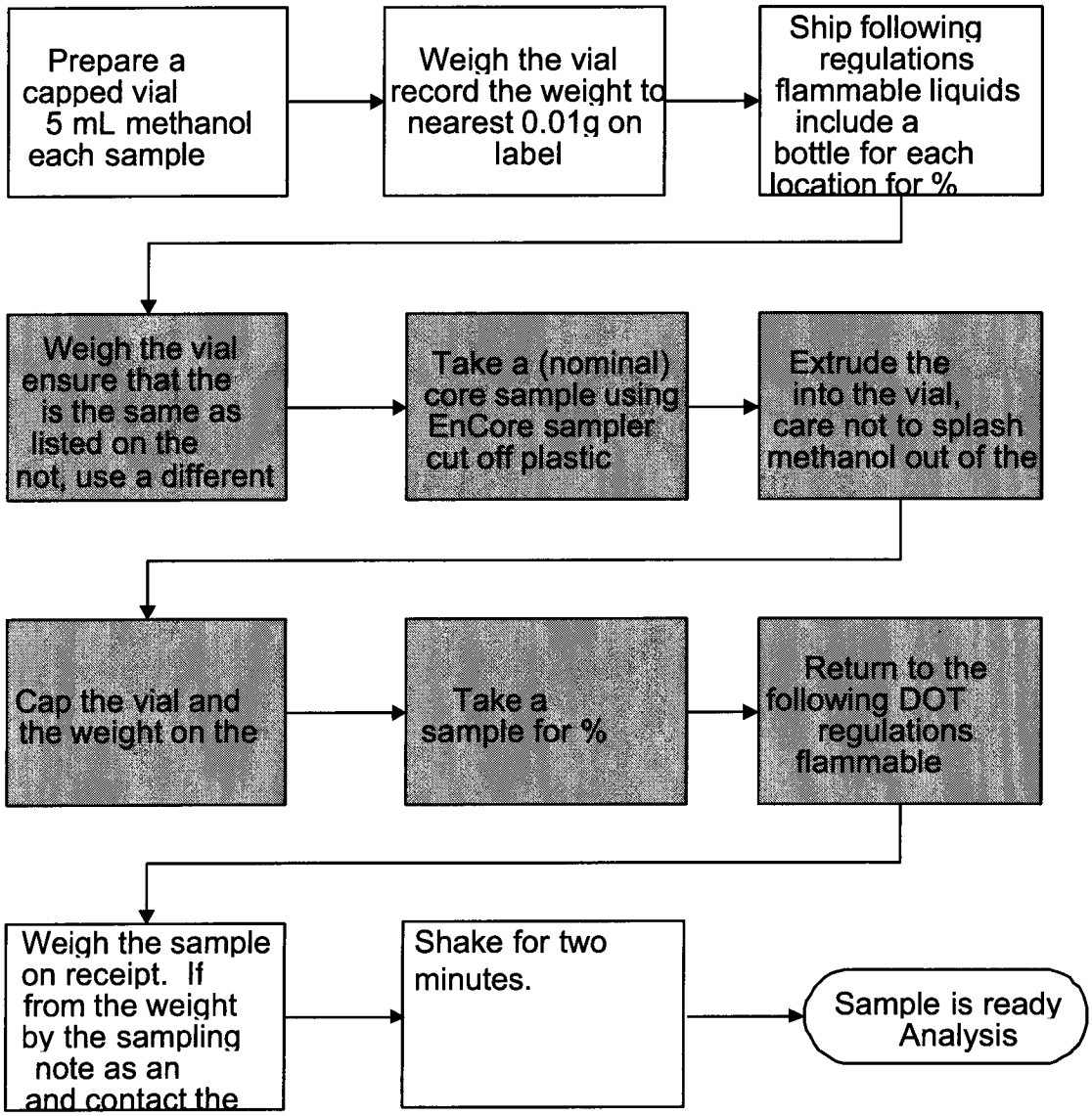
8.9. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.10. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

EnCore procedure when low level is not required (field steps in gray)



Field methanol extraction procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Refer to the method 8000B section of this SOP, section 9, for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the Method 8000B section of this SOP, Section 10, for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For simultaneous determination of aromatic and halogenated volatiles, a single column is used and the PID and ELCD detectors are connected in series.

10.2.1. Refer to Table A-2, A-3 and A-4 for GC operating conditions. Additional operating instruction may be found in instrument manuals located in the laboratory.

10.3. Initial Calibration

10.3.1. Refer to Section 10 of the 8000B section of this SOP for details of initial calibration criteria.

10.3.2. Low-level soil samples must be purged at 40°C; therefore the calibration curve must also be purged at 40°C.

10.3.3. The low-level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector.

10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Calibration Verification

10.4.1. A mid level calibration standard is used for the calibration verification. The gases have 20 % D criteria rather than the 15% used for other analytes. For analytes not listed in Method 8021B, the CCV criteria is 50% D.

10.4.2. A calibration verification run is performed after every ten samples for this method.

10.4.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Analytical Sequence

11.2.1. The analytical sequence starts with an initial calibration of at least five points.

11.3. Confirmation

11.3.1. The PID and ELCD detectors are sufficiently selective that second column confirmation is not

always necessary. Requirements for second column confirmation should be decided in consultation with the client. If the PID and ELCD are used in series confirmatory information for many analytes can be gained by comparing the relative response from the two detectors.

The analytical sequence starts with an initial calibration of at least five points, or a 12 hour calibration that meets % difference criteria from an existing initial calibration.

11.4. Aqueous and Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.4.1. Check the pH of the sample remaining in the VOA vial prior to analysis. Samples are also checked for residual chlorine at this time.

11.4.2. Units, which sample from the VOA vial, should be equipped with a module, which automatically adds surrogate and internal standard solution, as needed, to the sample prior to purging the sample.

11.4.3. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.4.4. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5. *Low-Level Solids Analysis using discrete autosamplers Bulk Solids*

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.5.1. Do not discard any supernatant liquids. Mix the contents of the container.

11.5.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or40mL vial. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5 g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium level method described in section 11.7.

11.5.3. Place in Autosampler.

11.5.4. Add 5 mL of organic free water to each vial. Add surrogate/internal standard (and matrix spike solutions if required.) (See Tables A-5, A-6, A-7 and A-8.) Add directly to the sample from 11.6.2.

11.5.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.5.6. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect. If external standard calibration is used, samples with surrogate recovery below the control limit should be reanalyzed once to confirm matrix effect.

11.6. Methanol Extract Soils

11.6.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to section 12 of the 8000B section of this SOP.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the 8000B section of this SOP.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste streams produced by the method.

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

15.2.1.1. **Acidic material from the auto-sampler.** Waste stream must be collected and neutralized before discharge to a sewer system if the pH is less than 4.

15.2.1.2. **Methanol waste from rinses and standards**. Methanol waste is discarded as a flammable liquid .

15.2.1.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The waste coordinator handles crushing the vials and proper disposal.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B.

16.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996.

16.3. Laboratory Holding Blanks, NC-QA-0020

17. MISCELLANEOUS

17.1. TABLES

Table A-1

Standard Analyte List for Method 8021B

Compound	CAS No.	Reporting Limit, µg/L or µg/kg		
		Aqueous	Low Soil	Medium Soil
1,1,1,2-Tetrachloroethane	630-20-6	1.0		
1,1,1-Trichloroethane	71-55-6	1.0		
1,1,2,2-Tetrachloroethane	79-34-5	1.0		
1,1,2-Trichloroethane	79-00-5	1.0		
1,1-Dichloroethane	75-34-3	1.0		
1,1-Dichloroethene	75-45-4	1.0		
1,1-Dichloropropene	563-58-6	1.0		
1,2,3-Trichlorobenzene	87-61-6	1.0		
1,2,3-Trichloropropane	96-18-4	1.0		
1,2,4-Trichlorobenzene	120-82-1	1.0		
1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	50
1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0		
1,2-Dibromoethane(EDB)	106-93-4	1.0		
1,2-Dichlorobenzene	95-50-1	1.0		
1,2-Dichloroethane	107-06-2	1.0		
1,2-Dichloropropane	78-87-5	1.0		
1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	50
1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
1,3-Dichloropropane	142-28-9	1.0		
1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
2,2-Dichloropropane	590-20-7	1.0		
2-Chloroethyl vinyl ether	110-75-8	5.0		
2-Chlorotoluene	95-49-8	1.0		
4-Chlorotoluene	106-43-4	1.0		
Acetone	67-64-1	10		
Benzene	71-43-2	1.0	1.0	50
Benzyl Chloride	100-44-7	5.0		
Bromobenzene	108-86-1	1.0		
Bromochloromethane	74-97-5	1.0		
Bromodichloromethane	75-27-4	1.0		
Bromoform	75-25-2	1.0		
Bromomethane	74-83-9	1.0		
Carbon Tetrachloride	56-23-5	1.0		
Chlorobenzene	108-90-7	1.0		

Table A-1

Standard Analyte List for Method 8021B

Compound	CAS No.	Reporting Limit, µg/L or µg/kg		
		Aqueous	Low Soil	Medium Soil
Chlorodibromomethane	124-48-1	1.0		
Chloroethane	70-00-3	1.0		
Chloroform	67-66-3	1.0		
Chloromethane	74-87-3	1.0		
cis-1,2 Dichloroethene	156-59-4	1.0		
cis-1,3-Dichloropropene	10061-01-5	1.0		
Dibromomethane	74-95-3	1.0		
Dichlorodifluoromethane	75-71-8	1.0		
Ethyl Benzene	100-41-4	1.0	1.0	50
Freon 113	76-13-1	1.0		
Hexachlorobutadiene	87-68-3	1.0		
Isopropylbenzene	98-82-8	1.0		
MEK (2-butanone)	78-93-3	5.0		
Methyl tert-butyl ether (MTBE)	1634-04-4	1.0		
Methylene Chloride	75-09-2	5.0		
MIBK (4-methyl-2-pentanone)	108-10-1	5.0		
Naphthalene	91-20-3	2.0	2.0	250
n-butylbenzene	104-51-8	1.0		
n-Propylbenzene	10306501	1.0		
p-Isopropyltoluene	99-87-6	1.0		
sec-Butylbenzene	135-98-8	1.0		
Styrene	100-42-5	1.0		
tert-Butylbenzene	98-06-6	1.0		
Tetrachloroethene	127-18-4	1.0		
Toluene	108-88-3	1.0	1.0	50
trans-1,2-Dichloroethene	156-60-5	1.0		
trans-1,3-Dichloropropene	10061-02-6	1.0		
Trichloroethene	79-01-6	1.0		
Trichlorofluoromethane	75-69-4	1.0		
Vinyl Chloride	75-01-4	1.0		
Xylenes (total)	1330-20-7	1.0	1.0	50

Table A-2
Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles

Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3
Surrogate and Internal Standard Concentrations

Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Final concentration µg/L (µg/kg)
Combined aromatic and halogenated volatiles IS/SS	Fluorobenzene (SS)	50	1	10
	1,4-Dichlorobutane (SS)	50	1	10
	a,a,a-Trifluorotoluene	50	1	10

It may be necessary to select different surrogates in order to minimize sample interferences. 1-chloro-4-fluorobenzene and 4-chlorotoluene are fairly well resolved from analytes listed in this SOP. However 4-chlorotoluene may sometimes be requested as a target analyte. Other surrogates that may be considered, and issues associated with their use are:

Bromochloromethane: Elutes very close to chloroform and cis-1, 2-dichloroethene on the 502.2 column. May be a target analyte.

1-Chloro-2-fluorobenzene: Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2

Bromofluorobenzene: Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.

2-Bromo-1-chloropropane: May coelute with 1,1,2-trichloroethane

Table A-4

Concentrations for LCS and MS/MSD compounds

Standard	Components	Working Solution µg/mL	Spike amount µL (5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
Halogenated	Chlorobenzene	20	5	20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20
Combination aromatic / Halogenated	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. This Appendix may also to be applied when discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica North Canton sample extraction SOPs. (CORP-OP-0001NC)

1.2. Table B-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

1.3. At client request, this method may also be used for the analysis of PCBs (Aroclors) in combination with pesticides, although these are normally analyzed following method 8082, as described in Appendix C of this SOP. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.

1.4. The associated LIMS method code is QJ (8081A).

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation is by internal or external standard methods.

3. DEFINITIONS

3.1. Refer to the TESTAMERICA North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-0025, Cleanup SOP.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Using hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds should be prepared in a hood.

5.4. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.5. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table B-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table B-3 for details of calibration standards.

7.3. Surrogate Standards

7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to tables B-5 and B-6 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

7.4.1. A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Refer to Table B-2 for recommended details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns.

10.3. Column Degradation Evaluation

10.3.1. Before any calibration runs, either initial or 12 hour, The column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

10.3.2. If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

10.4. Replacement of the injection port liner or the glass wool.

10.5. Cutting off a portion of the injection end of a capillary column.

10.6. Replacing the GC column.

10.7. Initial Calibration

10.7.1. Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

10.7.2. Refer to Table B-7 for the initial calibration analytical sequence.

10.7.3. The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

10.7.4. The surrogate calibration curve is calculated from the Individual AB mix. Surrogates in the other calibration standards are used only as retention time markers. If there are resolution problems, then the A and B mixes may be analyzed separately.

10.7.5. For multi-component pesticides:

10.7.5.1. A five-point calibration is used for multi-component pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to section 12.3 for guidance on which option to use.

10.7.6. A full 5 point calibration for any of the multi-component analytes is analyzed.

10.8. 12-hour Calibration Verification

10.8.1. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

10.8.2. At a minimum, the 12-hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multi-component analytes.

10.8.3. The retention time windows for any analytes included in the 12-hour calibration are updated.

10.9. Continuing Calibration

10.9.1. The AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. The continuing calibration standard need not include multi-component analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

10.9.2. A mid level calibration standard is used for the continuing calibration.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP No. CORP-OP-0001NC.

11.3. Cleanup

11.3.1. Cleanup procedures are described in SOP No. NC-OP-0025.

11.4. Suggested gas chromatographic conditions are given in Table B-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table B-7.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multi-component Analytes

12.2.1. Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Quantitation of Multi-component Analytes

12.3.1. Use 3-10 major peaks (or total area for quantitation) as described in section 10.4.4, initial calibration of multi-component analytes.

12.3.2. If there are no interfering peaks within the envelope of the multi-component analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.3.2.1. Multiple peak option

12.3.3. This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find

the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

12.3.4. Chlordane may be quantitated either using the multiple peak option (12.3.1.1) total area option (12.3.1.2.) or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

12.4. Total area option

12.4.1. The total area of the standards and samples may be used for quantitation of multi-component analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multi-component pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

12.5. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

12.6. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.7. Calculation of Column Degradation/% Breakdown (%B)

Equation 9

12.7.1. $$DDT\,\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

Where:

A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

Equation 10

$$Endrin\,\%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

Where:

A_{EK}, A_{EA}, and A_{E} = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

14.1 Refer to Section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8081A

17. MISCELLANEOUS

17.1. Modifications from Reference Method - None

17.2. TABLES

Table B-1
Standard Analyte List and Reporting Limits for Method 8081A

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD-ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzilate	0.1	3.3	100
Kepone[1]	1.0	33	1000

[1] Kepone is sometimes requested for analysis by method 8081A. However kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative.
Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects. The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table B-2
Recommended Conditions for Method 8081A

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.32mm id, 0.5µm
Column 2	Rtx-35 30m x 0.32 mm id, 0.5µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3
Calibration Levels ng/mL for Method 8081A

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane[3]	5	10	25	50	100	200
γ-Chlordane[3]	5	10	25	50	100	200
Multi-component Standards						
Chlordane (Technical)	20	50	100	200	500	
Toxaphene	200	500	1000[5]	2000	5000	
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.
[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

Table B-4	
Column Degradation Evaluation Mix ng/mL for Method 8081A	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg for Method 8081A			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	33.3	200
Aldrin	0.20	33.3	200
Heptachlor	0.20	33.3	200
Dieldrin	0.50	33.3	500
Endrin	0.50	33.3	500
4,4'DDT	0.50	33.3	500
Tetrachloro-m-xylene (Surrogate)	0.20	33.3	200
Decachlorobiphenyl (Surrogate)	0.20	33.3	200

Table B-6		
LCS/Matrix Spike and Surrogate Spike levels for TCLP µg/L or µg/kg for Method 8081A		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B-7
Suggested Analytical Sequence for Method 8081A

Initial Calibration

Solvent blank (optional)	
Primer if needed	
Breakdown Mix	
Individual mix AB	All levels
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	

Any multi-component analytes

[1] A five-point curve for any of the multi-component analytes may be included
If Aroclors are included, a 5 point calibration for Aroclor 1016/1260 should be included with the initial calibration and a single point for the other Aroclors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

Note: The initial primer is used if the instrument has been idle for a period of time.

12 -Hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica sample extraction SOP (CORP-OP-0001NC). The PCBs are determined and quantitated as Aroclor mixes.

1.2. Table C-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

1.2.1.1. Note: SW-846 method 8082 provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

1.3. The associated LIMS method code is QH (8082).

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

3.1. Refer to the TestAmerica North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP NC-OP-0025.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts including, but not limited to Mercury, Tetrabutylammonium sulfite (TBA), Sulfuric Acid Cleanup, Silica Gel Cleanup, and Florisil® Cleanup of semivolatile organic compound extracts based on SW846 Methods 3660B, 3665A, 3630C, and 3620B. These cleanup procedures are included in SOP NC-OP-0025.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and

clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.4. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table C-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.2. Refer to Table C-3 for details of calibration standards.

7.3. Surrogate Standards

7.3.1. Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to Table C-4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1 Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Initial Calibration

10.2.1. Refer to Table C-6 for the initial calibration analytical sequence.

10.2.2. The response for each Aroclor will be calculated by the procedures described in the general method for GC analysis, with the following modifications.

10.2.3. A minimum five-point calibration of all Aroclors is generated. The average response factor is used to quantitate Aroclors. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector.

10.2.4. The high and low standards for the initial five-point calibration of 1016 / 1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration the extract must be diluted and reanalyzed.

10.2.4.1. NOTE: For Ohio VAP, Aroclor 1268 may be analyzed. In order to meet project specific reporting limits, a lower concentration standard may be added to the calibration curve.

10.2.5. If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor may be used for the five point calibration rather than the 1016 / 1260 mix.

10.2.6. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.7. The following is used for the quantitation of all Aroclors. The same quantitation option must be used for standards and samples.

10.2.7.1. Multiple peak option.

10.2.7.2. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks.

10.3. 12-Hour Calibration

10.3.1. The 12 -our calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration.

10.3.2. At a minimum, the 12-hour calibration includes analysis of the Aroclor 1260 / 1016 mix.

10.3.3. Other Aroclors are included in the daily calibration check.

10.3.4. The retention time windows for any analytes included in the daily calibration and CCVs are updated.

10.3.5. For this method samples must be bracketed with successful calibration verification runs.

10.4. Calibration verification standards

10.4.1. The Aroclor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

10.4.2. A mid level standard is used for the calibration verification.

11. PROCEDURE

11.1. Refer to the Method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP CORP-OP-0001NC.

11.3. Cleanup

11.3.1. Cleanup procedures are described in SOP NC-OP-0025.

11.4. Suggested gas chromatographic conditions are given in Table C-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table C-6.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Aroclors

12.1.1. Retention time windows are used for identification of Aroclors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.1.2. A clearly identifiable Aroclor pattern serves as confirmation of single column GC analysis. Dual column confirmation may be used for specific program requirements or by client request.

12.2. Quantitation of Aroclors

12.2.1. Use 3-10 major peaks or total area for quantitation

12.2.2. If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1248 and 1232 is present, then only the predominant Aroclor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative. If well separated Aroclor patterns are present, and then multiple Aroclors may be quantitated and reported.

12.3. Second column confirmation of Aroclors will only be performed when requested by the client. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence.

12.4. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits, or if one is <10%.

12.4.1. NOTE: For Ohio VAP samples and DoD projects, all surrogates must meet acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

13.2. Method detection limits (MDL) are determined for all Aroclors.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Refer to Section 15 of the 8000 section of this SOP

15.2. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional

information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8082

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. TABLES

Table C-1
Standard Analyte list and Reporting Limits for Method 8082

Compound	Reporting Limit, µg/L or µg/kg		
	Water	Soil	Waste
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table C-2
Instrumental Conditions for Method 8082

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C-3
Calibration Levels ng/mL for Method 8082

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[1]
Aroclor 1016/1260	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1242[2]	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1221 +1254[2]	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1232[2]	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1248[2]	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1262	0.05	0.1	0.2	0.5	1.0	2.0
Aroclor 1268	0.05	0.1	0.2	0.5	1.0	2.0
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[2] Aroclors may be quantitated within the range 100 to 2000 ng/mL (4000ng/mL if the level 6 1016/1260 standard is included). If the Aroclor is more concentrated, it must be reanalyzed at a dilution.

Table C-4
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis µg/L or µg/kg for Method 8082

	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table C-5
Michigan Analyte List and Reporting Limits[1] for Method 8082

	Reporting Limit		
Compound	water (µg/L)	soil (µg/Kg)	
Aroclor-1016	0.2	330	
Aroclor-1221	0.2	330	
Aroclor-1232	0.4	330	
Aroclor 1242	0.2	330	
Aroclor-1248	0.2	330	
Aroclor-1254	0.2	330	
Aroclor-1260	0.2	330	

[1] Reporting Limits are only for samples performed under the Michigan program

Table C-6
Suggested Analytical Sequence for Method 8082

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
8	Aroclor 1242	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
9	Aroclor 1248	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
10	Aroclor 1221/1254	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
11	Aroclor 1268 or 1262	An Initial Calibration includes Levels 1-5. A midpoint is used as CCV.
12	ICV	
13-32	Sample 1-20 (or as many samples as can be analyzed in 12 hours	
33	Aroclor 1016/1260	Level 3

etc

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12-hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1260 / 1016 mix. Mid level standards of any other Aroclors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

1.1. This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides in extracts prepared by SOP NC-OP-0031NC. The herbicides listed in Table D1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met.

1.2. The associated LIMS method code is QS.

2. SUMMARY OF METHOD

2.1. This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

3.1 Refer to the TestAmerica North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Sulfur may interfere and may be removed by the procedure described in SOP# NC-OP-0025.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A Ni_{63} electron capture detector is required.

6.2. Refer to Table D2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP for general information on reagents and standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1 Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control

samples (LCS), and matrix spikes (MS).

9.2. Refer to Table D-3 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Calibration standards are prepared from purchased standards in the methyl ester form.

10.3. The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

10.4. Refer to Table D-2, for details of GC operating conditions.

11. PROCEDURE

11.1. Refer to the Method 8000B section of this SOP for procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

11.3.1. The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

11.4.1. The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.2. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence.

11.4.3. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.4. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. Gas Chromatography

11.5.1. Chromatographic conditions are listed in Table D-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration

standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

12.3. A routine 10X dilution occurs on final extracts for all samples. Due to a QuantIMS limitation, the dilution factor field in QuantIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in QuantIMS.

13. METHOD PERFORMANCE

13.1. The EPA for this method has not published multiple laboratory performance data. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Refer to the method 8000B section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

17.2.1. The calibration procedure has been changed to require esterification of the calibration standards

17.3. TABLES

Table D-1				
Standard Analyte List for Method 8151A				
Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL 10 mL	10	
Low-level Soil without GPC	50 g	10 mL	10
High-level soil / waste	1 g	10 mL	10

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D-1.

Table D-2	
Instrumental Conditions for Method 8151A	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5um
Column 2	DB-1701 or Rtx-1701
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table D-1.

The reporting limits listed in Table D-1 will be achieved with these calibration levels and a 20-fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table D-3
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg[1] for Method 8151A

	Aqueous	Soil	Waste
2,4-D	40	400	20000
Silvex	10	100	5000
2,4,5-T	10	100	5000
2,4-DB	40	400	20000
Dalapon	20	200	10000
DCAA (surrogate)	40	400	20000
Dicamba	20	200	10000
MCPP	4000	40000	200000
MCPA	4000	40000	200000
Dichloroprop	40	400	2000
Pentachlorophenol	5	50	2500
Dinoseb	6	60	300

[1] LCS, MS and SS spikes are as the free acid.

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of the concentration and **tentative** identification of extractable petroleum (diesel range) hydrocarbon mixes in waters, wastewaters, soils, and sludges.

1.2. This SOP is based on SW-846 Method 8015B, Modified, Revision 3, December 1996.

1.3. The associated LIMS method codes are HS (8015 MOD) and KI (8015B).

2. SUMMARY OF METHOD

2.1. This method provides gas chromatographic conditions for detection and identification of total petroleum hydrocarbons. Prior to the use of this method, appropriate sample preparation techniques are used.

2.2. An aliquot of the prepared sample is injected into a gas chromatograph (GC) and compounds in the effluent are detected by a flame ionization detector (FID).

2.3. The laboratory carbon range for Ohio VAP projects is C10-C20 and C20-C32. The laboratory carbon range for BUSTR Projects is C10-C20 and C20-C34.

3. DEFINITIONS

3.1. Refer to the glossary in the TESTAMERICA North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP.

7.2. The petroleum hydrocarbons are purchased from a chemical supplier when available. When no chemical supplier is available, the fuels are purchased from public sources.

7.3. The OVAP and BUSTR standard is a commercially prepared standard containing alkanes from C10-C34.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1. Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. MS/MSD recoveries are calculated from a Diesel calibration.

9.3. Surrogates

9.3.1. Because of the nature of the TPH analysis, whereas certain petroleum mixtures can override the C9 (Nonane) surrogate. The C9 (Nonane) surrogate recoveries are advisory. Re-extraction due to surrogate recoveries is determined by analyst judgement.

9.3.1.1. NOTE: Ohio VAP rules require reanalysis when surrogate recoveries are outside of control limits.

10. CALIBRATION AND STANDARDIZATION

Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.1. Recommended Instrument Conditions

10.1.1. Hydrogen carrier gas - flow rate 5 - 6 mL/min

10.1.2. Detector gas mixture - air hydrogen mixture in a 10:1 ratio, air 80 - 120 mL/min, hydrogen 8 -12 mL/min

10.1.3. Temperature Program - refer to Table F1 Appendix

10.1.4. Injection volume - 1 µL

10.2. Initial Calibration

10.2.1. Analyze a five point Diesel calibration standard referring to the recommended instrument conditions. The calibration concentrations are 100, 200, 500, 1000, and 2000 ng/uL. A 5000ng/uL standard may be analyzed if needed. The retention time window of C10-C32 shall be used for the Diesel calibration. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector.

10.2.2. For Ohio VAP and BUSTR projects, the laboratory analyzes a five point calibration for the carbon range C10-C20. The concentrations are 60, 120, 240, 600 and 1200 ug/mL. In addition, a five point of the carbon range C20-C34 is also analyzed. The concentration ranges are 80, 160, 320, 800, and 1600 ug/mL.

10.3. Continuing Calibration

10.3.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.3.2. A mid-range standard of Diesel, C10-20, and C20-34 is used, as appropriate, for the CCV. The acceptance criteria is 15%.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

11.2.1. The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Analytical Sequence – Refer to Section 11 in the 8000B Section of this SOP.

11.4. Petroleum Hydrocarbon Identification and/or Fingerprinting

11.4.1. To identify the type of petroleum hydrocarbon, compare the chromatographic peak pattern to the patterns of known petroleum hydrocarbons analyzed under identical chromatographic conditions. Samples are quantified against diesel, but fingerprinting may be done when client requested.

11.4.2. Positive matching may not be possible, even using site-specific hydrocarbons. Degradation of the pattern can occur during environmental exposure of the fuel. See Table 2 for possible fingerprints.

11.4.3. Sample Quantification

11.4.4. Samples are quantified against the initial calibration of diesel or DRO on a single column.

11.4.5. The total height or area of the hydrocarbon is determined in the same manner used for the hydrocarbon standard.

11.4.6. If the amount of sample injected into the GC exceeds the working range of the calibration curve, an appropriate dilution is performed before reanalysis.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Nonane (C-9). The surrogate must be within QC criteria. Corrective action is only necessary if Nonane (C-9) is outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to Section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

16.2. Related SOP

16.2.1. CORP-OP-0001NC, Extraction of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, 8150, 8151, and 600 Series Methods

Table E-1
Suggested GC Temperature Program for TPH Analysis

Initial Temperature	40°C
Initial Hold Time	4 minutes
Temperature Program	10°C/minute
Final Temperature	280°C
Final Hold Time	10 minutes

Table E-2
Reporting Limits for TPH Analysis

Analyte	Reporting Limits		
	Water (µg/L)	Solids (mg/kg)	Waste Dilution (mg/kg)
TPH (as Diesel) or DRO	100	3.3	200
C10-C20 (OVAP & BUSTR)	60	2.0	
C20-C34 (OVAP & BUSTR)	80	2.3	
Fingerprint Compounds [1]			
Mineral Spirits	Kerosene	Motor Oil	
Hydraulic Oil	Jet Fuel	Stoddard Solvent	
DRO Spiking Solution			
Decane	Dodecane	Tetradecane	
Hexadecane	Octobecane	Eicosane	
Docosane	Tetracosane	Hexacosane	
Octacosane			

[1] This list represents most of the common petroleum hydrocarbons. The list may be expanded to include other petroleum hydrocarbons.

Title: ANALYSIS OF DISSOLVED GASES IN GROUNDWATER

[Modified Method RSK-175]

Approvals (Signature/Date):

Technology Specialist	Date	Health & Safety Coordinator	Date
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP NC-GC-0032, Rev 2, dated 02/01/07

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1. SCOPE AND APPLICATION

1.1 This document describes a procedure for the determination of dissolved gases in groundwater. The method is applicable to the preparation of water samples for the analysis of the headspace to quantify part-per-billion levels of dissolved gases in water samples.

1.2 The associated QuantIMs method code is MM.

1.3 This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A water sample is collected in the field in a 43-mL VOA vial with no headspace. Prior to analysis, the sample is transferred into a 22-mL serum vial with a crimp cap. Headspace is generated using UHP helium. The sample is loaded onto the headspace autosampler and analyzed by a Gas Chromatograph (GC) equipped with an FID detector.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. 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.

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with a sticker that reads "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and to a Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1 Sample Containers: 44 mL VOA vials, 22 mL crimp cap vials

6.2 Instrumentation

6.2.1 Column (FID) - Restek Rt-UPLOT; 30m, 0.53mm ID

6.2.2 Autosampler - Tekmar 7000 Headspace Autosampler

6.3 Syringes - 10µL - 5.0-mL gas tight syringes.

6.3 Data System – Chemstation for acquisition and Target™ for data processing.

7. REAGENTS AND STANDARDS

7.1. Gas cylinders of ultrahigh purity helium, hydrogen, and nitrogen.

7.2 Calibration Standards: The primary standard is purchased through Scott Specialty Gases. The FID calibration standard is composed of nominally 1% (mole basis) methane, ethane, ethene, acetylene, and propane.

7.3 The calibration levels are achieved by using an SGE syringe to inject different amounts of the standard into a 22-mL vial that contains 18 mL of deionized water and 4 mL of headspace.

7.4 Laboratory Control and Initial Calibration Verification Samples: The LCS and ICV are fortified to the concentration of a standard near the midpoint of the calibration curve.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples are collected in the field in a 43-mL VOA vial. The samples are preserved with 1:1 HCl to a pH of less than 2. Acid converts inorganic carbon to carbon dioxide; therefore, acid should not be added if carbon dioxide is a compound of interest. Care should be taken that no headspace is present when capping the vials. Samples are maintained at a temperature of 4°C ± 2°C and should be analyzed within 14 days of collection.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents, the same processes, and the same personnel.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit, with the exception of common laboratory contaminants.

9.2.1.1. The common laboratory contaminant for this method is Methane. Methane can be present up to five times the reporting limit.

9.2.2. Corrective Action for Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS from an independent source must be processed with each preparation batch. The hydrocarbon LCS is fortified to the concentration of a standard near the midpoint of the calibration curve. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. Corrective Action for LCS

9.3.2.1. If any analyte is outside established control limits, the system is out of control and corrective action must occur.

9.3.2.2. The only exception is if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.2.3. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair is processed at client request for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's)

may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. Corrective action for MS/MSDs

9.4.2.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.2.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as DIL (diluted out).

9.4.2.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.4.2.4. If client program requirements specify to confirm matrix interference's, repreparation and reanalysis of the MS/MSD may be necessary.

9.5. Control Limits

9.5.1. Control limits are established by the laboratory as described in SOP NC-QA-0018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOP NC-QA-0021 and CA-Q-S-006.

9.6.2. MDLs are easily accessible via LIMs (QC Browser program).

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration –Establish an initial calibration curve using the concentrations noted in the table below.

Compound	Cal Level 1	Cal Level 2	Cal Level 3	Cal Level 4	Cal Level 5	Cal Level 6	Cal Level 7
Volume Injected (uL)	0.65	1.3	50	200	600	1000	1000
Methane	0.24	0.47	18	73	219	364	1456
Ethane	0.45	0.89	34	137	410	683	2732
Ethene	0.42	0.83	32	127	383	638	2552
Acetylene	0.36	0.77	30	118	355	592	2368

Concentrations are in µg/L unless noted otherwise.

10.1.1. For each analyte, calculate the mean calibration factor from analyses of the calibration solutions.

10.1.2. Calculate the standard deviation (SD) and relative standard deviation (RSD) from each mean.

10.1.3. The percent RSD average of all analytes must be ≤ 30%.

10.1.4. 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.

10.1.5. If the analyst can document that a level is not valid because of an injection or instrument problem confined to that run, 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.

10.1.6. 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 external standard methods, the average amount of analyte recovered is less than 70% or greater than 130% of the expected value.

10.1.7. 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.

10.1.8. 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.

10.1.9. 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

10.2 Continuing Calibration Verification – Analyze a continuing calibration verification at the beginning of each 24-hr analytical window. The CCV is fortified to the concentration of a standard near the midpoint of the calibration curve. The %D between the CCV CF and the calibration average CF for each analyte must be less than or equal to 30%.

10.3 Initial Calibration Verification - An ICV must be analyzed run following the acquisition of the five-point initial calibration. The ICV is fortified to the concentration

of a standard near the midpoint of the calibration curve. The calibration factor (CF) for the ICV must be within 30%D of that from the initial calibration.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by the QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Analysis

11.3.1. Sample Analysis (GC-FID) - Remove the samples from the refrigerator and allow to come to room temperature. Pour the sample into a 22-mL vial and immediately seal with a crimp cap, taking care to avoid headspace. Insert a 22-gauge needle into the septum. Using a 5-mL gastight syringe, inject 4 mL of UHP helium into the sample. The helium forces out an equal amount of sample through the 22-gauge needle to create a headspace volume of 4 mL. Withdraw the needle and syringe from the vial and load the sample onto the Tekmar headspace autosampler. The autosampler allows the sample's water and headspace phases to equilibrate at 40°C. 100 µL of the sample headspace are injected directly onto the GC column where the target compounds, if present, are detected by FID. Acquire the data and process on Target. The instrument operating conditions are outlined below.

<u>Recommended GC Conditions</u>

Gas Flows:	53.2 mL/min
Carrier (Helium):	20 mL/min
Oven Program:	50°C for 2.1 minutes

<u>Recommended FID Conditions</u>

FID Temp:	200°C
Hydrogen Flow:	40 mL/min
Air Flow:	450 mL/min
Nitrogen Makeup:	45 mL/min

11.4. Analytical Documentation

11.4.1. Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.4.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.4.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.4.4. Sample results and associated QC are entered into LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1 Calibration Factor for GC-FID

$$\mathbf{CF_\chi = A_\chi / C_\chi}$$

where:

CF_χ	=	Calibration factor of compound χ
A_x	=	Peak height or area
C_x	=	Concentration of target analyte χ in sample (ug/L)

12.2 Percent Difference for Calibration Factors

$$\mathbf{\% D = (CF_{ave} - CF_c / CF_{ave}) \times 100}$$

where:

CF_{ave}	=	Average CF for an analyte from the initial calibration
CF_c	=	CF for an analyte from current check standard

12.3 Relative Standard Deviation

$$\mathbf{\%RSD = (SD / CF_{ave}) \times 100}$$

where:

CF_{ave}	=	Average CF for an analyte from the initial calibration
SD	=	Standard Deviation of average CFs for a compound

12.4 Sample Concentration in water

$$C_\chi = (A_\chi / CF_{ave}) \times DF$$

where:

C_χ	=	Concentration of target analyte χ in sample (ug/L)
A_χ	=	Peak area of analyte χ
CF_{ave}	=	Average calibration factor for an analyte from the calibration
DF	=	Dilution factor

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

15.2.1. There are no waste streams produced when this method is carried out

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. RSK SOP-175, Revision 0, August 11, 1994.

16.1.2. TestAmerica North Canton Quality Assurance Manual (QAM), current version.

16.1.3. TestAmerica Corporate Safety Manual, M-E-0001 and TestAmerica North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021 and CA-Q-S-006

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.6. Standards and Reagents, NC-QA-0017

16.2.7. Acceptable Manual Integration Practices, CA-Q-S-002

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The reporting limit for all analytes is 1 ug/L.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.1.3. Compound Constants

Table 1: Compound Constants

Compound	Molecular Weight (g)
Methane	16
Ethane	30
Ethene	28
Acetylene	26

APPENDIX I
EXAMPLE CALCULATION

(Calculations Based on 22°C at 754 mmHg – Molar Equivalent .04099)

(Level 6) 1000 ul of 10,000 ppmv – 18 mL H_20

$$\frac{10{,}000\,ulCH_4}{LN_2} \times \frac{1LCH_4}{1{,}000{,}000\,ulCH_4} \times \frac{.04099\ molesCH_4}{1LCH_4} \times \frac{16\,gCH_4}{1moleCH_4} \times \frac{.001\,LN_2}{.018\,LH_20} \times \frac{1{,}000{,}000\,ugCH_4}{1gCH_4} = \frac{6.5584}{.018} = \frac{364\,ugCH_4}{LH_20}$$

Reviewed/Revised by CF1, 11/19/03
Reviewed/Revised by CF2, 2/24/04
Reviewed/Revised by CF3 9/20/04
Revised by CF4, 8/29/05
Reviewed 2/3/06
Revised by CF5, 1/9/07
Reviewed 3/19/07

Title: Method 8290 and TO-9A – Polychlorinated Dioxins and Furans by HRGC/HRMS Sample

[Method 8290]

Approvals (Signature/Date):

01/17/2008	1/17/08
Patrick Rainey Date Technical Manager	Joe Schairer Date Health & Safety Manager / Coordinator
Pamela Schemmer 1/17/08	for K. Buechler 1/17/08
Pamela Schemmer Date Quality Assurance Manager	Karla Buechler Date Laboratory Director

This SOP was previously identified as SAC-ID-0005.

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1. SCOPE AND APPLICATION

1.1. This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is also described. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits and other pertinent information.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques.

2.2. If interferences are encountered, the method provides selected cleanup procedures to aid the analyst in their elimination. A simplified analysis flow chart is show in Figure 1.

2.3. A specified amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still-bottom, fuel oil, chemical reactor residue, air sample (QFF, PUF or XAD media) or fish tissue, is spiked with a solution containing specified amounts of each of nine isotopically (13C) labeled PCDDs/PCDFs listed in Table 2. The sample is then extracted according to a matrix-specified extraction procedure. The extraction procedures are: a) toluene Soxhlet (or equivalent) extraction, for soil, sediment, fly ash samples, aqueous sludges, and solid air matrices (XAD, QFF, PUF); b) methylene chloride liquid-liquid extraction for water samples; c) dilution of a small sample aliquot

in solvent for wastes/chemical products; and d) toluene (or hexane/methylene chloride) Soxhlet (or equivalent) extraction for fish tissue.

2.4. If interferences are present, extracts may be cleaned as described below. The extracts are submitted to an acid and/or base washing treatment and dried. Following a solvent exchange step, the residue is cleaned up by column chromatography on acid/base silica, acid alumina and carbon on silica. The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 20 µL of a tetradecane solution containing 100 pg/µL of each of the two recovery standards 13C-1,2,3,4-TCDD and 13C-1,2,3,7,8,9-HxCDD (Table 2) to the concentrated eluate. The former is used to determine the percent recoveries of tetra- and penta-chlorinated PCDD/PCDF internal standards while the latter is used for the determination of hexa-, hepta- and octa-chlorinated PCDD/PCDF internal standard percent recoveries. Upon client approval, less final volume can be used to decrease detection limit and more final volume can be used to decrease severe interferences.

2.5. One to two µL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).

2.6. The identification of ten of the 2,3,7,8-substituted congeners (Table 3), for which a 13C-labeled standard is included as a spiked compound, is based on their elution at their exact retention time (-1 to +3 seconds from the respective internal or recovery standard signal) and simultaneous detection of the two most abundant ions in the molecular ion region. All other identified PCDD/PCDF congeners are identified by their relative retention times based on the daily CCV standard , and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.

2.7. Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.

3. DEFINITIONS

3.1. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 2) that contain from one to eight chlorine atoms. The seventeen 2,3,7,8-substituted PCDDs and PCDFs are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.

3.2. Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.

3.3. Isomer: Defined by the arrangement of chlorine atoms within a homologous series. For example, 2,3,7,8-TCDD is a TCDD isomer.

3.4. Congener: Any isomer of any homologous series.

3.5. Surrogate Standards: A 13C-labeled analog or mixture of analogs that are added to the sample collection media prior to shipment to the field.

3.6. Internal Standard: An internal standard is a 13C-labeled analog of a congener chosen from the compounds listed in Table 3. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

3.7. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The 13C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while 13C-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. 13C-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.8. High-Resolution Concentration Calibration Solutions (Table 5): Tetradecane solutions containing known amounts of the 17 2,3,7,8-substituted PCDDs and PCDFs, a minimum of nine internal standards (13C-labeled PCDDs/PCDFs), and two carbon-labeled recovery standards; the set of five solutions is used to determine the instrument response of the unlabeled analytes relative to the internal standards and of the internal standards relative to the recovery standards.

3.9. Sample Fortification Solution (Table 2): A solution (isooctane or toluene) containing the nine internal standards, which is used to spike all samples before extraction and cleanup.

3.10. Recovery Standard Solution (Table 2): A tetradecane solution containing the two recovery standards, which is added to the final sample extract before HRGC/HRMS analysis.

3.11. Field Blank: A portion of a sample representative of the matrix under consideration, which is used to assess potential PCDD/F contribution from sampling and transport.

3.12. Rinsate: A portion of solvent used to rinse sampling equipment. The rinsate is analyzed to demonstrate that samples were not contaminated during sampling.

3.12.1.1. GC Column Performance Check Mixture: A tetradecane solution containing a mixture of selected PCDD/PCDF standards including the first and last eluters for each homologous series, which is used to demonstrate continued acceptable performance of the capillary column (i.e., ≤ 25 percent valley separation of 2,3,7,8-TCDD from all the other 21 TCDD isomers) and to define the homologous PCDD/PCDF retention time windows.

3.13. Performance Evaluation Materials (PEM's): Representative sample portions containing known amounts of certain unlabeled PCDD/PCDF congeners (in particular the ones having a 2,3,7,8-substitution pattern). Representative interferences may be present. PEMs may be obtained from the EPA EMSL-LV or other sources and submitted to potential contract laboratories, which must analyze these and obtain acceptable results before being awarded a contract for sample analyses (see IFB Pre-Award Bid Confirmations). PEMs may also be included as unspecified ("blind") quality control (QC) samples in any sample batch submitted to a laboratory for analysis.

3.14. Relative Response Factor: Response of the mass spectrometer to a known amount of a native analyte relative to a known amount of an internal standard, or a known amount of internal standard to a known amount of a recovery standard.

3.15. Sample Re-extraction: Extraction of another portion of the sample followed by extract cleanup and extract analysis.

3.16. Extract Reanalysis: Instrument analysis by HRGC/HRMS of another aliquot of the final extract.

3.17. Tuning (Mass Resolution Check): Standard method used to demonstrate a static resolving power of 10,000 minimum (10 percent valley definition).

3.18. Method Calibration Limits: For a given sample size, a final extract volume, and the lowest and highest concentration calibration solutions, the lower and upper calibration limits delineate the region of quantification for which the HRGC/HRMS system was calibrated with standard.

3.19. Matrix Spike Fortification Solution: Solution used to prepare the laboratory control sample, matrix spike, and matrix spike duplicate samples. It contains all unlabeled analytes listed in Table 5. The solution also contains all internal standards used in the sample fortification solution per the method.

3.20. Definitions of other terms used in this SOP may be found in the glossary of the Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Re-use of glassware is to be minimized to avoid the risk of contamination.

4.4. Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.

4.5. A high-resolution capillary column (60m DB-5) is used to resolve as many PCDD and PCDF isomers as possible. However, no single column is known to resolve all isomers. The DB-225 column is used for the quantitation of 2,3,7,8-TCDF when the quantitated value for 2,3,7,8-TCDD on the DB-5 column is greater than the Target Detection Limit (TDL), or 1/2 the lower calibration limit.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported immediately to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns or Requirements

5.1.1. Hearing protection must be worn when using mechanical systems to grind fish or tissue samples.

5.1.2. When dissecting crawfish abdomens with a scalpel, cut from the hand holding the abdomen toward the tail (away from you).

5.1.3. Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.

5.1.4. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

5.1.5. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.6. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.7. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.8. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. 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 (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.
Benzene	Flammable Toxic Carcinogen	PEL: 1 PPM TWA ; 5 PPM 15 MIN. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhileration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.
Cyclohexane	Flammable Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.
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.
Iso-octane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
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.
Potassium Hydroxide	Corrosive Poison	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 the severity of exposure. Symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures.
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 (1)	Corrosive Oxidizer Dehydra-dator	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.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

6.1.1. The GC must be equipped for temperature programming. All required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. The use of a moving needle injection port is also acceptable. When using the method described in this protocol, a 2-µL injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 µL). 1 µL injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times on a given HRGC/HRMS/DS.

6.1.2. Gas Chromatograph/Mass Spectrometer (GC/MS) Interface - The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel® or equivalent ferrules are recommended.

6.1.3. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.

6.1.4. Data System - A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data for a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral

peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

6.2. GC Column

6.2.1. Due to poor separation of 2,3,7,8-TCDF from other TCDF isomers on the 60 m DB-5 column, a 30M DB-225 is used to quantitate 2,3,7,8-TCDF. This column is used when the quantitated value for 2,3,7,8-TCDF is greater than the TDL.

6.2.2. In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended. At the beginning of each 12-hour period during which samples are analyzed and after tuning, acceptable compound separation on the GC column must be demonstrated through the analysis of a column performance check solution. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

6.3. Miscellaneous Equipment and Materials

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.3.1. Nitrogen evaporation apparatus with variable flow rate.

6.3.2. Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3.3. Centrifuge.

6.3.4. Water bath, equipped with concentric ring covers and capable of maintaining temperature control within ± 2°C.

6.3.5. Stainless steel or glass containers large enough to hold contents of one-pint sample containers.

6.3.6. Drying oven.

6.3.7. Stainless steel spoons and spatulas.

6.3.8. Pipettes, disposable, Pasteur, 150 mm long x 5 mm ID.

6.3.9. Pipettes, disposable, serological, 10 mL, for the preparation of the carbon column specified in Section 7.1.

6.3.10. Reacti-vial, 2 mL, silanized clear glass.

6.3.11. Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.

6.3.12. Separatory funnels, 250 mL.

6.3.13. Separatory funnels, 1000 mL.

6.3.14. Teflon® boiling chips (or equivalent) washed with DCM before use.

6.3.15. Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon® stopcock.

6.3.16. Adapters for concentrator tubes.

6.3.17. Glass fiber filters.

6.3.18. Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125 mL flask.

6.3.19. Continuous liquid-liquid extractor.

6.3.20. All-glass Soxhlet apparatus, 500 mL flask.

6.3.21. Soxtherm extraction apparatus (or equivalent), including glass thimble holders, glass beakers, and gaskets.

6.3.22. Glass funnels, sized to hold 170 mL of liquid.

6.3.23. Desiccator.

6.3.24. Turbo evaporator

6.3.25. Rotary evaporator with a temperature controlled water bath.

6.3.26. High speed tissue homogenizer, equipped with an EN-8 probe or equivalent.

6.3.27. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

Note: Re-use of glassware should be minimized to avoid the risk of contamination. All glassware that is re-used must be scrupulously cleaned as soon as possible after use, applying the following procedure:

6.4. Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by adsorption on the glassware surface.

6.4.1. Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

6.4.2. After detergent washing, glassware should be immediately rinsed with acetone, toluene, hexane, and then methylene chloride.

6.4.3. Do not kiln reusable glassware in an oven as a routine part of cleaning. Kilning may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated kilning of glassware may cause the formation of active sites on the glass surface that will irreversibly adsorb PCDDs/ PCDFs.

6.4.4. Immediately prior to use, Soxhlet (or equivalent) extraction glassware should be pre-extracted with toluene for a minimum of 3 hours. Note: Accelerated extractors such as the Soxtherm can use a shorter cleaning cycle which exhibits subsequent extractions free of cross contamination and interferences.

7. REAGENTS AND STANDARDS

7.1. Column Chromatography Reagents

7.1.1. Silica Gel - Kieselgel 60 or equivalent, activate for 1 hour at 184°C before use. Store at 130°C in covered flask.

7.1.2. Acid Alumina - ICN or equivalent, activated as necessary.

7.1.3. Basic Alumina - ICN or equivalent. No activation required.

7.1.4. Granular carbon/silica gel - Mix 3.6 g granular carbon and 16.4 g activated silica gel; (alternatively, prepare carbon/silica gel (5%/95%); i.e., combine 5 g precleaned carbon with 95 g silica gel). Store at room temperature in a Teflon ® lined covered jar. The first LCS prepared with a new batch of column packing material is the quality control check of the packing materials. Refer to historical control limits before accepting the new batch of material.

7.1.5. 44% H_2SO_4 /silica gel - Mix 24 mL conc. H_2SO_4 and 56 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.1.6. 33% NaOH/silica gel - Mix 34 mL 1N NaOH and 67 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.2. Reagents

7.2.1. Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.

7.2.2. Potassium hydroxide, ACS grade, 20 percent (w/v) in distilled water.

7.2.3. Distilled water demonstrated to be free of interferents

7.2.4. Potassium carbonate, anhydrous, analytical reagent.

7.2.5. Silica gel.

7.2.6. Solution for breaking emulsions: Slowly add 1.0L of reagent grade NaOH solution to a 2.0L NaOH container, containing 1.0L of DI H2O, and leave the container in secondary containment with the lid off. Warning: The solution will begin to heat so let the solution stand until equilibrium is met and the solution is at room temperature. When this process is complete, the solution will then be ready for use in the samples.

7.2.7. Precleaned Ottawa sand (minimum 4 hours toluene Soxhlet Extracted).

7.2.8. Canola Oil (for tissue extraction only).

7.3. Desiccating Agent

7.3.1. Sodium sulfate, granular, anhydrous.

7.4. Solvents

7.4.1. High-purity, distilled-in-glass or highest available purity: Methylene chloride, hexane, benzene, methanol, tetradecane, isooctane, toluene, cyclohexane, and acetone.

7.5. All calibration, daily internal standard, daily clean up recovery standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.5.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.6. Calibration Solutions

7.6.1. High-Resolution Concentration Calibration Solutions (Table 5) - Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 16) PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (0.5 pg/µL) and the highest for the octachlorinated congeners (2000 pg/µL).

7.6.2. Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

7.6.3. Store the calibration solutions in appropriate containers and at room temperature in the dark.

7.7. GC Column Performance Check Solution

7.7.1. This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The 13C-2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/µL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution for the DB-5 column.

7.7.2. For the DB-225 column, the column performance check solution contains a series of TCDF isomers in addition to the 2,3,7,8-TCDF. The solution is injected and evaluated at the start of each analytical sequence on the DB-225 column to ensure that 2,3,7,8-TCDF is resolved from its closest eluting isomers with a baseline-to-valley ratio of ≤ 25%. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution on for the DB-225 column.

7.8. Field Surrogate Solution (air matrices)

7.8.1. This solution contains one 37Cl labeled analog (for Method TO-9/TO-9A) or one 37C and four 13C labeled analogs (for Method 0023) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.9. Sample Fortification Solution (Internal Standard)

7.9.1. This isooctane (or toluene) solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that 13C-OCDF is not present in the solution.)

7.10. Recovery Standard Solution

7.10.1. This tetradecane solution contains two recovery standards (13C-1,2,3,4-TCDD and 13C-1,2,3,7,8,HxCDD). An appropriate volume of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

7.11. Preparation and QC of PUF material

7.11.1. The PUF material is purchased pre-cut.

7.11.2. The PUFs are rinsed by Soxhlet with toluene (or other appropriate solvent) for a minimum of 16 hours and air dried for a minimum of 2 hours in a contaminant-free area.

7.11.3. One PUF from the rinsed batch is randomly selected to be the QC sample for the batch.

7.11.4. The PUF is loaded into a pre-cleaned Soxhlet extractor charged with toluene.

7.11.5. The 1613/8290 daily internal standard solution is spiked into the PUF and it is extracted for a minimum of 16 hours.

7.11.6. The Soxhlet extract is recovered and processed according to section 11.4.

7.11.7. The batch of PUF is considered acceptable if no target analytes are detected at or above the laboratory or project specific reporting limit.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire

contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See SAC-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. Grinding or blending of fish samples.

8.6.1. If not otherwise specified by the client, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3- to 5-mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the client. If so requested by the client, the above whole fish requirement is superseded.

Warning: Hearing protection must be worn when grinding samples.

8.7. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.8. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C).

8.9. Soil, Sediment or Paper Sludge (Pulp) Percent Moisture Determination.

The percent moisture of soil or sediment samples showing detectable levels (see note below) of at least one 2,3,7,8-substituted PCDD/PCDF congener is determined according to the following recommended procedure.

Generally, depending on sample availability, a 5-10 g sample, weighed to three significant figures, is used for % solids determination. The sample is then dried to constant weight at 110°C± 10 in an adequately ventilated oven. Weigh the dried solid to

three significant figures. Calculate and report the percent moisture on the appropriate form. Do not use this solid portion of the sample for extraction, but instead dispose of it as hazardous waste.

$$\text{Percent Moisture} = \frac{\text{Weight of wet soil - Weight of dry soil}}{\text{Weight of wet soil}} \times 100$$

8.10. Fish Tissue Lipid Content Determination

The percent lipid of fish samples is determined as follows:

Concentrate the extract from Section 11.3.5 on a rotary evaporator until constant weight is attained. The percent lipid is calculated using the following expression:

$$\text{Percent lipid} = \frac{\text{Weight of residue from extraction (in g)}}{\text{Weight of fish tissue portion (in g)}} \times 100$$

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, Ottawa sand, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. If the accompanying samples are aqueous, use distilled water as a matrix. Take the method blank through all steps detailed in the analytical procedure.

9.1.2. Use Ottawa sand as the method laboratory matrix when solids are extracted. Use a mixture of Ottawa sand and canola oil as the matrix when tissues are extracted. Take the method blank through all steps detailed in the analytical procedure.

9.1.3. The method blank must be spiked prior to extraction with the same amount of 13C-labeled internal standards as added to samples.

9.1.4. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

9.1.4.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.4.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.4.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.5. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy QA-003-SAC for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance can be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

9.3.5. Analyze the MS and MSD samples as described in Section 11.

9.3.6. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.7. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.5. Field Blanks

9.5.1. Each batch of samples may contain a field blank sample of nominally uncontaminated soil, sediment or water that is to be processed for analysis.

9.5.1.1. Weigh a 10-g portion or use 1 L (for aqueous samples) of the specified field blank sample and add the appropriate amount of internal standard to yield 100 pg/µL in the final extract.

9.5.1.2. Extract by using the procedures described in Section 11.3. As applicable, add the appropriate amount of recovery standard to yield 100 pg/µL in the final extract. Analyze a 1-2 µL aliquot of the concentrated extract.

9.5.1.3. Calculate the concentration of 2,3,7,8-substituted PCDDs/PCDFs and the percent recovery of the internal standards.

9.6. Rinsate Samples

9.6.1. In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

9.6.2. The rinsate sample must be processed like a regular sample.

9.6.2.1. Take a 100-mL (± 0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add the appropriate amount of internal standard to yield 100 pg/μL in the final extract.

9.6.3. Using appropriate methods, concentrate to approximately 10 mL.

9.6.4. Just before analysis, add the appropriate amount of recovery standard to yield 100 pg/μL in the final extract. Reduce the volume to a final volume of 20 μL, as necessary. No column chromatography is required.

9.6.5. Analyze an aliquot following the same procedures used to analyze samples.

9.6.6. Report percent recovery of the internal standard and the presence of any PCDD/PCDF compounds in pg/mL of rinsate solvent.

9.7. Surrogate/Clean Up Recovery Standard

9.7.1. A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up recovery standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of internal standard during both extraction and cleanup.

9.8. Internal Standards

9.8.1. Internal standards must be spiked into all samples, QC samples, and included in all calibrations.

9.8.2. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine internal standards.

9.8.3. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.9. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample portions.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION AND STANDARDIZATION

Calibration and Standardization requires a check of mass resolution (tuning), a check of chromatographic resolution, a verification of switching times (i.e. descriptors), and a calibration curve verification.

10.1. Tuning (Mass Resolution Check)

10.1.1. The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

10.1.2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.

10.1.3. By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). Verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).

10.1.4. Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760).

The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 5) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

10.2. Performance Checks

10.2.1. At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 3 (HRCC-3) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. (Note: A HRCC-2 or HRCC-4 may be acquired to meet the requirement of #2 above. This is to provide documentation of consistency for varying concentration levels, and to meet NELAC requirements). A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. The mass resolution check will be taken at the beginning and completion of an analytical sequence. An analytical sequence may contain one or more 12 hour periods.

10.2.1.1. Method blanks or solvent blanks are used to demonstrate that the analytical system is free of contamination after the analysis of calibration standards or high level samples. The blank must demonstrate that the system has returned to appropriate background levels prior to continued analysis.

10.2.2. At a minimum, the ions listed in Table 6 for each of the five SIM descriptors must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes closely to the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for 13C-HxCDF and 13C-HpCDF rather than M+2 and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6.

10.2.2.1. The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted native PCDDs/PCDFs, must be verified by comparison with second-source standard solutions.

10.3. Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Section 10.5) does not meet the required criteria listed in Section 10.6.

10.3.1. Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.

10.3.2. Tune the instrument with PFK.

10.3.3. Inject 1 or 2 µL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 6.1.3. The total cycle time must be ≤ 1 second. This is analyzed prior to a calibration curve to set descriptor windows only and may not otherwise be documented. The laboratory must not analyze samples until it is demonstrated and documented that the criterion listed in Section 13.1 is met.

10.3.3.1. Select the injection volume based upon the expected target analyte concentration, or expected matrix interferences.

10.3.3.2. The same injection volume must be used for all samples, QC, and standards.

10.3.4. By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 1 or 2-µL portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.

10.3.4.1. The total cycle time for data acquisition must be ≤ 1 second. The total cycle time includes the sum of all dwell times and voltage reset times.

10.3.4.2. Acquire SIM data for all the ions listed in the five descriptors of Table 6.

10.3.4.3. The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).

10.3.4.4. The ratio of integrated ion current for the ions belonging to the 13C labeled internal and recovery standards must be within the control limits stipulated in Table 9.

NOTE: Section 10.4.3 requires that ion ratios be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

10.3.5. For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

10.3.5.1. Referring to Table 10, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate internal standards (Table 5) and the nine RRFs for the labeled 13C internal standards [RRF(m); m=18 to 26] relative to the two recovery standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{is}}{Q_x \times A_{is}} \qquad RRF(m) = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs}}$$

Where:

A_x = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for unlabeled PCDDs/PCDFs,

A_{is} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled internal standards,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled recovery standards,

Q_{is} = quantity of the internal standard injected (pg),

Q_{rs} = quantity of the recovery standard injected (pg), and

Q_x = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF (n) and RRF (m) are dimensionless quantities; the units used to express Qis, Qrs, and Qx must be the same.

10.3.5.2. Calculate the RRF(n)s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = (1/5)\sum_{j=1}^{5} RRF_j(n)$$

Where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 10), and j is the injection number (or calibration solution number; j = 1 to 5).

10.3.5.3. The relative response factors to be used for the determination of the concentration of total isomers in a homologous series (Table 10) are calculated as follows:

10.3.5.3.1. For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 10.3.5.2.

NOTE: The calibration solutions do not contain 13C-OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the [M+6]+ ion of 13C-OCDF from the [M+2]+ ion of OCDD (and [M+4]+ from 13C-OCDF with [M]+ of OCDD). Therefore, the RRF for OCDF is calculated relative to 13C-OCDD.

10.3.5.3.2. For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RRF}(k) = (1/t)\sum_{n=1}^{t} RRF_n$$

Where:

k = 27 to 30 (Table 10), with 27 = PeCDF;
28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,
t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

10.3.5.4. Relative response factors [RRF(m)] to be used for the determination of the percent recoveries for the nine internal standards are calculated as follows:

$$RRF(m) = \frac{A_{is}^{m} \times Q_{rs}}{Q_{is}^{m} \times A_{rs}}$$

$$\overline{RRF}(m) = (1/5)\sum_{j=1}^{5} RRF_j(m)$$

Where:

m =	18 to 26 (congener type)
j =	1 to 5 (injection number),
A_{is}^{m} =	sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard (m = 18 to 26),
A_{rs} =	sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given internal standard (m = 18 to 26),
Q_{rs} & Q_{is}^{m} =	quantities of, respectively, the recovery standard (rs) and a particular internal standard (m) injected (pg),
RRF(m) =	relative response factor of a particular internal standard (m) relative to an appropriate recovery standard, as determined from one injection, and
$\overline{RRF}$(m) =	calculated mean relative response factor of a particular internal standard, as determined from the five initial calibration injections (j).

10.4. Criteria for acceptable calibration

The criteria listed below for acceptable calibration must be met before sample analysis is performed.

10.4.1. The percent relative standard deviations for the mean response factors [RRF(n)

and RRF(m)] from the 17 unlabeled standards must be ≤ 20 percent, and those for the nine labeled reference compounds must be ≤ 30 percent.

10.4.2. The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

10.4.3. The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 10.4.1 is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (Section 10.6) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

10.5. Routine Calibration (continuing calibration check)

Routine calibrations must be performed at the beginning of (following a successful tune and GC column performance check) and after a 12 hour period. The routine calibration initiates the 12 hour clock during which samples may be subsequently analyzed. The last sample in the sequence must be injected within 12 hours of the routine calibration, followed by the analysis of a closing calibration check. An acceptable closing calibration check standard may be used to initiate the next 12 hour analysis sequence when consecutive acquisition sequences occur. The ending mass resolution check shall be performed after the closing calibration check of an analysis acquisition sequence or after the final bracketing standard when consecutive 12 hour acquisition sequences are run.

10.5.1. Inject 1 or 2 µL of the concentration calibration solution HRCC-3 containing 10 pg/µL of tetrachlorinated congeners, 50 pg/µL of penta-, hexa-, and heptachlorinated congeners, 100 pg/µL of octachlorinated congeners, and the respective internal and recovery standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.3 through 6.2, determine and document an acceptable calibration as provided in Section 10.6.

10.6. Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken, including recalibration if needed.

10.6.1. The measured RRFs [RRF(n)] for the unlabeled standards obtained during the opening continuing calibration must be ± 20 percent of the mean values established during the initial calibration (Section 10.3.5.)

10.6.1.1. The bracketing continuing calibration must be ± 20% of the average RRF calculated from the initial calibration.

10.6.1.1.1. If the target compounds in the ending standard are less than or equal to ± 20% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the unlabeled isomers.

10.6.1.1.2. If the target analytes are greater than ± 20% but less or equal to ±25% and the samples are non-detect, the data is acceptable and this anomaly is documented. If these isomers are greater than ± 20% but less or equal to ±25% and are positive, an average RRF of the initial and ending daily standard is calculated and used to quantitate the concentration of the affected congener, and the anomaly is documented.

10.6.1.1.3. If the percent deviation of unlabeled compounds exceeds ± 25%, a new initial calibration is initiated within 2 hours following the analysis of the samples. Otherwise, reanalyze all sample extracts with positives for the failed target compounds.

10.6.2. The measured RRFs [RRF(m)] for the labeled standards obtained during the opening continuing calibration must be less than or equal to ± 30 percent of the mean values established during the initial calibration (Section 10.1.5).

10.6.2.1. The bracketing continuing calibration must be ± 30% of the average RRF calculated from the initial calibration.

10.6.2.1.1. If the labelled compounds in the ending standard are less than or equal to ±30% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the labeled isomers.

10.6.2.1.2. If the internal standard analytes are greater than ± 30% but less or equal to ±35%, an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener.

10.6.2.1.3. If the percent deviation of labeled compounds exceeds ± 35%, reanalyze samples if adversely impacted.

10.6.3. The ion-abundance ratios (Table 9) must be within the allowed control limits.

10.6.4. If either criteria in Sections 10.6.1 or 10.6.2 are not met, additional samples

may not be analyzed. Sample data collected must be evaluated for usability. Narrate any reported data from the analytical sequence. If the ion-abundance ratio criterion is not satisfied, refer to the note in Section 10.3.4.4 for resolution.

10.6.5. If either one of the above criteria (Sections 10.6.1 and 10.6.2) cannot be satisfied, the entire initial calibration process (Section 10.3) must be repeated.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Extraction

11.3.1. Internal Standard Addition. A 2000 pg aliquot of the internal standard mixture is added to all samples, regardless of sample size. As an example, for 13C-2,3,7,8-TCDD, a 10-g soil sample requires the addition of 2000 pg of 13C-2,3,7,8-TCDD to give the requisite fortification level.

11.3.2. Sludges. Paper Pulp Sludges are generally air-dried and ground. Because of the drying procedure, a Dean-Stark water separator is optional for extraction. Extraction is generally done by Soxhlet with 200-300 mL of toluene (150mL of toluene using the Soxtherm or equivalent).

11.3.2.1. Non-Paper Pulp Sludges are extracted with 200-300 mL of toluene (150mL of toluene using the Soxtherm or equivalent).

11.3.2.2. Extract the sample for a minimum of 16 hours (2 hours using the Soxtherm or equivalent). Cool the sample, filter the toluene extract, if needed, through a glass-fiber filter or equivalent into a round-bottom flask. Rinse the filter with 10 mL toluene, and combine the extract and rinsate. Concentrate the combined solutions to near dryness on a rotary evaporator at 50°C. Use of an inert gas to concentrate the extract is also permitted. Proceed with Section 11.6 as necessary.

11.3.3. Still-Bottom/Fuel Oil. All organic liquids, fuel oils, and solids that will dissolve in a solvent and are treated as a solvent dilution will be dissolved in 1-2 mL of an appropriate solvent; then diluted with 5-10 mL of hexane if no acid wash is required. Spike with appropriate Internal Standards and proceed with Section 11.6 as necessary.

11.3.4. Fly Ash. Extract fly ash samples by jar shake with hydrochloric acid before Soxhlet (or Soxtherm or equivalent) extraction. Weigh 2-10g of sample aliquot into a clean glass jar. Add 1.0mL of the internal standard mixture with 2 mL of acetone. Add 150 mL of 1N hydrochloric acid and shake for 4 hours. If the sample reacts violently with acid, then allow the sample to equilibrate for 4 hours with no shaking. The contents of the jar are then filtered through a glass fiber filter. The solids are Soxhlet extracted for 16 hours (2 hours using the Soxtherm or equivalent). The aqueous filtrate is extracted with 100 mL of toluene and twice with 100 mL of hexane. Concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50°C. Proceed with Section 11.6 as necessary.

Note: As an option, a Soxhlet/Dean Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.3.5. Solids/Tissues. Add anhydrous sodium sulfate to the soil sample portion in a ratio of 2 to 1 (e.g. 20 g sodium sulfate to 10 g of sample) and mix thoroughly with a stainless steel spatula. After breaking up any lumps, place the soil/sodium sulfate mixture in the Soxhlet apparatus on top of a glass-wool plug (the use of an extraction thimble is optional). Add 200 to 250 mL toluene to the Soxhlet apparatus and reflux for 16 hours (150mL of toluene and 2 hour extraction using the Soxtherm or equivalent). The solvent must cycle completely through the system at least five times per hour. Proceed with Section 11.6.

Note: The matrix for the tissue method blank consists of 9g of pre-cleaned Ottawa sand and 1g of canola oil.

11.3.6. Preparation of stationary source samples. See SOP SAC-ID-0009.

11.4. Preparation of crawfish samples

11.4.1. Prior to dissecting the crawfish, rinse the animal with reagent water.

11.4.2. Isolate the crawfish abdomen by manually pulling the abdomen away from the cephalothorax.

11.4.3. Using forceps, hold the abdomen down and make a ventral incision of the cuticle along the full length of the abdomen midline using either dissection

scissors or a scalpel blade. If using a scalpel blade, cut from the hand toward the tail.

11.4.4. Hold open the abdomen midline incision and remove the abdominal flexor muscle (tail meat) with forceps and place in a weighing boat.

11.4.5. If any of the anterior portion of the abdominal flexor muscle remained in the cephalothorax, remove it with forceps and combine with the muscle portion from 11.4.4. The combined portion should yield 3-6 grams of meat.

11.4.6. With a spatula, remove a portion of the hepatopancreous from the cephalothorax. An attempt should be made to recover as much of the hepatopancreous as possible. This should yield a few hundred milligrams of tissue. (Test yielded approximately 0.5-0.8 g wet weight of hepatopancreous from medium sized crawfish.)

11.4.7. Combine the hepatopancreous with the abdominal flexor muscle tissue. (Tests yielded approximately 5 g total wet weight of combined tissue per crawfish specimen using 12-18 g animals.)

11.4.8. Repeat steps 11.4.1 through 11.4.6 with additional crawfish specimens and composite the tissue until sufficient tissue mass is acquired for sample preparation and analysis as specified in the appropriate laboratory SOPs. (The composited tissue may be required for more than just the Dioxin analysis described in this SOP).

11.4.9. Record the composited tissue mass collected.

Note: The laboratory may elect to perform a combination of Dioxin/Furans and PCBs, in which case a 20 gram aliquot of the sample will be extracted and split prior to extract cleanup.

11.4.10. Homogenize the composited tissue and proceed with removal of a sample aliquot for extraction and analysis as directed in Section 11.4.11.

11.4.11. Weigh approximately a 10 gram aliquot into an extraction thimble and record the sample weight on the extraction sheet.

11.4.12. Place the extraction thimble containing the sample in a pre-cleaned Soxhlet extractor charged with 200-220 mL of fresh toluene and boiling chips (150mL toluene and 2 hour extraction using the Soxtherm or equivalent).

11.4.13. Proceed with the extraction according to section 11.3.5.

Note: If a % lipid determination is required, a tared round bottom flask is used. There is no solvent keeper added prior to extract concentration by rotary evaporation and the sample is reduced to dryness and reweighed according to section 8.11. The sample is then redissolved in hexane and process to section 11.6.2.

11.4.14. Aqueous Samples. Weigh the sample bottle. Also, mark the water meniscus on the side of the 1-L sample bottle for later determination of the exact sample volume. Pour the entire sample (approximately 1-L) into a 2-L separatory funnel.

11.4.14.1. Add 100 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel. The internal standard mixture is first mixed in 2 mL of acetone then it is added to the sample in the separatory funnel. Each aliquot of spike mixture is added similarly. Extract the sample by shaking the funnel for two minutes with periodic venting.

Warning: DCM will rapidly build up pressure. The funnel should vented initially after a few shakes.

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 volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. Extraction is repeated two additional times with methylene chloride.

Warning: Separatory funnel extraction is a high risk activity. Analyst will wear a face shield over safety glasses/goggles for this extraction. Alternatively, the extraction can take place behind a closed hood sash.

11.4.14.2. Determine the original sample volume by re-weighing the sample bottle, or filling the sample bottle to the mark with water and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11.4.14.3. Dry extract with sodium sulfate: Place glass wool in a precleaned filter funnel. Rinse glass wool with DCM and load funnel with DCM-rinsed Na_2SO_4. Pour extract through Na_2SO_4 to remove water. Rinse Na_2SO_4 with fresh DCM and collect in round bottom flask.

11.4.14.4. Transfer the extract to a 500-mL round-bottom, add approximately 100 µL of tetradecane and concentrate on a rotary evaporator or TurboVap.

11.4.15. A continuous liquid-liquid extractor may be used in place of a separatory funnel when experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered when using a separatory funnel.

11.4.15.1. Add 60 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface.

11.4.15.2. Transfer the solvent to the extractor.

11.4.15.3. Repeat the sample bottle rinse with an additional 50- to 100-mL portion of methylene chloride and add the rinsate to the extractor.

11.4.15.4. Add 200 to 500 mL methylene chloride to the distilling flask, add sufficient reagent water to ensure proper operation, and extract for 24 hours.

11.4.15.5. Allow to cool, then detach the distilling flask.

11.4.16. Filter/PUF Samples

11.4.16.1. Place the glass sleeve containing the PUF and the Quartz Fiber Filter into the pre-cleaned Soxhlet extractor charged with toluene.

11.4.16.2. Add 2 mL (4000 pg) of 1613/8290 Internal Standard solution to all samples and QC.

11.4.16.3. Add 50 mL of 1613/8290 Native Spike to the LCS.

11.4.16.4. Extract the samples and QC for a minimum of 16 hours.

11.4.16.5. Concentrate the extract from the round bottom flask with hexane and adjust the volume.

11.4.16.6. Transfer the extract from the round bottom flask with hexane and adjust the volume.

11.4.16.7. Split the extract 50:50 for analysis and archive.

11.4.16.8. Proceed to Section 11.6.

11.4.17. Wipe Extractions

11.4.17.1. Jar Shake Method

11.4.17.1.1. Place a pre-cleaned wipe in a French Square jar. This will be the method blank aliquot. Place an additional pre-cleaned wipe in yet another French Square. This will be the LCS aliquot.

11.4.17.1.2. Transfer each wipe sample and all accompanying liquid into separate French Square jars or appropriate sized containers (Note: If the container used to deliver the wipe to the laboratory can contain 100 ml of solvent, then the container can be used for the extraction).

11.4.17.1.3. Spike all samples, method blank and LCS with an appropriate amount of internal standard. Additionally, spike the LCS with the appropriate amount of native standard.

11.4.17.1.4. Add 100 ml of toluene to each jar and secure Teflon lined cap. Place the closed container onto the flatbed shaker and secure in place. Turn the shaker on such that the shaking motion is aggressive enough to move the liquid through the wipe. Shake for 4 hours.

11.4.17.1.5. Filter each sample through a filter funnel with a glasswool plug. Capture the sample into a 500 ml round bottom flask. Add approximately 100 µL of tetradecane and concentrate to approximately 100 µL on a rotary evaporator or TurboVap.

11.4.17.1.6. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Insure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.4.17.1.7. Upon completion of the rinsing, cap the test tube and shake vigorously. Take ½ of each sample (or an

appropriate amount deemed from the client or other method) and transfer to a culture tube. Archive the remaining sample for future use.

11.4.17.1.7.1. If only one analysis is required, then ½ of the sample is archived and the other half is analyzed.

11.4.17.1.7.2. If three analyses are required, then 1/3 is archived 1/3 is used for one test and 1/3 is used for the third test. For additional analyses, adjust the fractions accordingly.

11.4.17.2. Soxhlet Method

11.4.17.2.1. Place a pre-cleaned wipe in a precleaned Soxhlet extraction apparatus. This will be the method blank aliquot. Place an additional pre-cleaned wipe in yet another precleaned Soxhlet extraction apparatus. This will be the LCS aliquot.

11.4.17.2.2. Transfer each wipe sample and all accompanying liquid into separate precleaned Soxhlet extraction apparatus.

11.4.17.2.3. Spike all samples, method blank and LCS with an appropriate amount of internal standard. Additionally, spike the LCS with the appropriate amount of native standard.

11.4.17.2.4. Charge the Soxhlet with approximately 300 mL toluene and completely assemble the Soxhlet apparatus. Turn the temperature to the appropriate setting to cycle the toluene. Cycle for 16 hours.

11.4.17.2.5. After cycling is complete, turn heat off and allow glassware to cool. Remove round bottom flask. Add approximately 100 µL of tetradecane and concentrate to approximately 100 µL on a rotary evaporator or TurboVap.

11.4.17.2.6. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional

amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Insure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.4.17.2.7. Upon completion of the rinsing, cap the test tube and shake vigorously. Take ½ of each sample (or an appropriate amount deemed from the client or other method) and transfer to a culture tube. Archive the remaining sample for future use.

11.4.17.2.7.1. If only one analysis is required, then ½ of the sample is archived and the other half is analyzed.

11.4.17.2.7.2. If three analyses are required, then 1/3 is archived 1/3 is used for one test and 1/3 is used for the third test. For additional analyses, adjust the fractions accordingly.

11.5. There are several useful methods to decrease or eliminate emulsion in aqueous samples when extracting with DCM. These methods may include stirring with a pipette to manually breakup the emulsions or to transfer the sample into centrifuge tubes and centrifuge at approximately 3000 RPM. The most useful method is to use a 1:1 $NaOH/H_2O$ solution to change the pH enough to disrupt the emulsion phase, which works 90% of the time. See section 7.2 for reagent preparation.

11.5.1. Breaking Emulsions:

11.5.1.1. Check the pH of the sample to verify that the pH is between 3 and 7. If the pH is greater than 7, consult the supervisor and client for instructions.

11.5.1.2. Pour approximately 100 mL of the 1:1 $NaOH:H_2O$ into a 1.0L AGB.

11.5.1.3. Drain the sample with the emulsion from the 2.0L separatory funnel into the 1L AGB and let it stand.

11.5.1.4. Empty the aqueous waste into the LLE waste drum.

11.5.1.5. Pour the solution with DCM back into the same 2.0L separatory funnel and drain the DCM phase through Na_2SO_4 into a 500mL round-bottom flask.

11.5.1.6. Empty the aqueous waste into the LLE waste drum.

11.5.1.7. Proceed with section 11.6.

11.6. Optional Extract Clean-Ups

For all samples which are not air media, spike 1.0 mL of the Cleanup Recovery Standard (CRS) prior to any cleanup into the round bottom flasks containing the samples and QC Extracts (See also Section 9.71)

11.6.1. Acid/Base Partitioning

11.6.1.1. Partition the extract in 50-125 mL of hexane against 40 mL concentrated H2SO4 in a separatory funnel. Shake for two minutes. Remove and discard the sulfuric acid layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

Warning: Shaking with a concentrated caustic is a high-risk activity. Analyst must wear a face shield over safety glasses/goggles, or the shaking must take behind a closed hood sash.

11.6.1.2. Partition the extract against 50 mL distilled H_2O. Shake for two minutes. Remove and discard the aqueous layer (bottom). If further cleanup is required, proceed to section 11.6.1.3

11.6.1.3. Partition the extract using 50 mL of 10 N NaOH. Shake for two minutes. Remove and discard the aqueous layer (bottom). Repeat the base washing until no color is visible in the bottom layer (perform a maximum of four base washings). Strong base is known to degrade certain PCDDs/PCDFs, contact time must be minimized. The NaOH partition is applied only as samples warrant it at the discretion of the analyst.

Warning: Shaking with a concentrated caustic is a high-risk activity. Analyst must wear a face shield over safety glasses/goggles, or the shaking must take behind a closed hood sash.

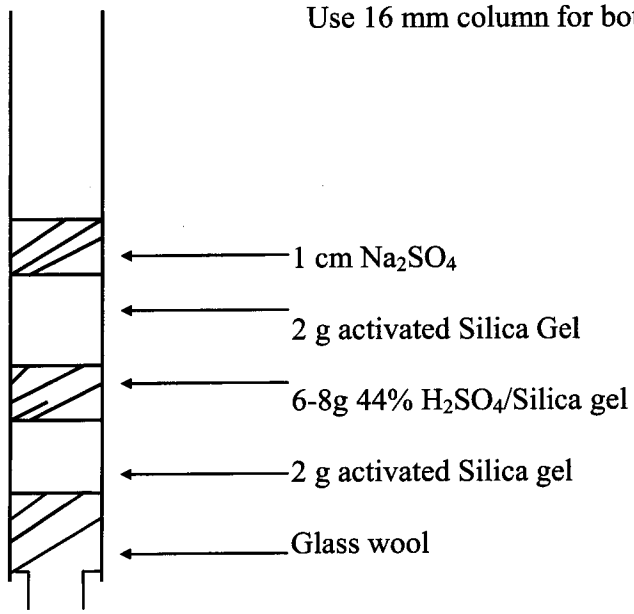
11.6.1.4. Partition the extract against 50 mL of distilled H_2O. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a funnel containing anhydrous

sodium sulfate and collect it in a round-bottom flask. Rinse the sodium sulfate with two 15-mL portions of hexane, add the rinsates to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (when applicable) are removed. (Use of blow-down with an inert gas to concentrate the extract is also permitted.) The DI H_20 partition is applied only as samples warrant it at the discretion of the analyst.

11.6.2. Silica Column Clean-Up "Exhibit A"

EXHIBIT A
IFB Column Clean-up

Use 20 mm column for top column
Use 16 mm column for bottom column*



*Upper and lower columns are piggy backed – See Exhibit B for lower column packing

11.6.2.1. Pre-rinse both columns with hexane - 20 mL Top and 20 mL Bottom.

11.6.2.2. Put one column above the other.

11.6.2.3. Add extract to the top column (silica column). Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.6.2.4. Elute 60 mL hexane directly onto acid silica column (upper piggy backed columns).

11.6.2.5. Discard upper column.

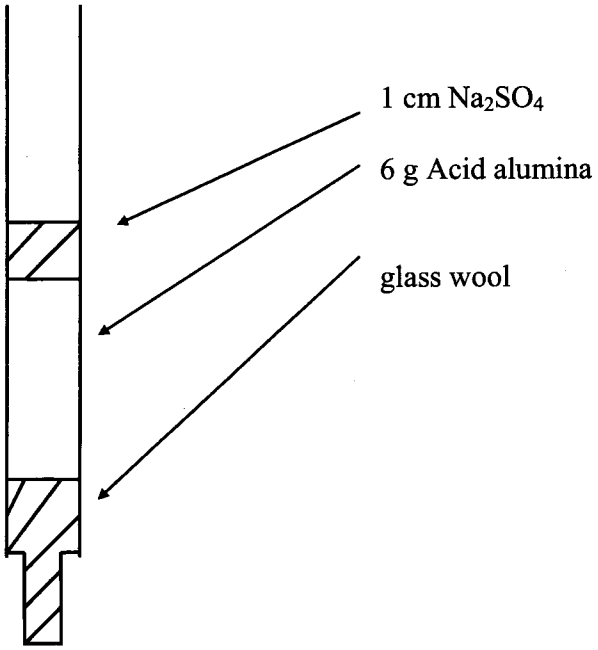
11.6.2.6. Elute lower column with 10 mL of 20% methylene chloride/hexane. Discard in proper waste stream.

11.6.2.7. Elute lower column with 30 mL of 65% methylene chloride/hexane. Save and collect in culture tube.

11.6.2.8. Proceed with additional cleanups as necessary.

11.6.3. Acid Alumina Column Clean-Up "Exhibit B"

Exhibit B
Acid Alumina Column Clean-up



11.6.3.1. Alumina activity may vary with the matrix or environmental conditions. Monitor internal standard and cleanup recovery standard recoveries in extract analysis. Low recoveries of cleanup recovery standard (CRS) may indicate loss of alumina activity. Assess stability of alumina activity and apply corrective action as appropriate (reactivate and reprofile). Note: a column profile should be done to show elution of all 2,3,7,8 substituted analogs so problems can be readily identified (see section 11.6.3.2 for profile technique).

11.6.3.2. Profile each vendor lot of activated alumina as corrective action for low internal standard and CRS recoveries dictate. If necessary, proceed as follows:

11.6.3.2.1. Set up and label 3 acid alumina columns.

11.6.3.2.2. Pre-rinse with 20 mL hexane.

11.6.3.2.3. Add 2 mL hexane spiked with internal standards and natives (spike amounts equivalent to those for LCS) with 2X2 mL hexane rinse of fractions.

11.6.3.2.4. Elute each column with 20 mL hexane. Collect and label these fractions.

11.6.3.2.5. Elute each column with 5 x10 mL methylene chloride/hexane at the appropriate v/v percent. Collect and label these fractions separately.

11.6.3.2.6. Elute each column with 10 mL of 100% methylene chloride. Collect and label these fractions. Reduce all fractions to final volume and add recovery standard.

11.6.3.3. Review data and select an elution scheme. Group the fraction from each solvent system as follows:

11.6.3.3.1. Pre-analyte fraction - consists of all eluent prior to elution of first target analytes.

11.6.3.3.2. Analyte fraction - consists of all that contain detectable levels of target analytes.

11.6.3.3.3. Post-analyte fraction - consists of all eluents after elution of the last target analyte.

11.6.3.4. Select the solvent system which best meets the following two conditions:

11.6.3.4.1. Pre-analyte fraction consists of 20mL hexane and no more than 20 mL mixed solvent.

11.6.3.4.2. Analyte fraction consists of no more than 20mL of mixed solvent and contains greater than 90% of all target analytes and greater than 80% of all internal standards.

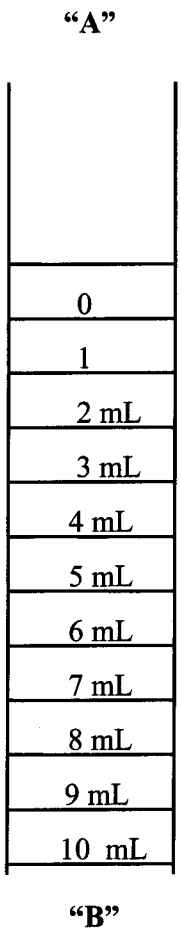
11.6.3.5. After selection of the appropriate solvent system and fractionation pattern, perform triplicate acid alumina cleanups on spiked hexane to ensure reproducibility of the fractionation pattern. Document each elution scheme.

11.6.3.6. Each subsequent batch of acid alumina used in the lab (from the same vendor lot) must be checked for stable activity.

11.6.4. Carbon Column Clean-up "Exhibit C"

11.6.4.1. Prepare an activated Carbon & Silica Gel column as described in EXHIBIT C, below.

Exhibit C
Carbon Column Clean-up
Special D2



- Cut off both ends of a 10 mL pipette, or use pre-cut column.
- Push a glasswool plug down to the mark.
- Add 1g of 5% activated carbon/silica and top with another glasswool plug.
- Pre-elute with 5 mL 1:1 MeCl2:cyclohexane. Direction "A"
- Turn over and pre-elute with 5 mL 1:1 $MeCl_2$:cyclohexane in direction "B".
- Discard pre-eluates.
- Dilute extract to 1 mL with hexane and transfer to the column in the "B" direction .
- Rinse sample vial onto the column with 2 X 2 mL 1:1 MeCl2:cyclohexane.
- Elute with 6mL 1:1 MeCl2:cyclohexane

  5mL 75:20:5 MeCl2:MeOH:Benzene
- Discard Evaluates
- After flipping column back to the "A" direction, the column is eluted with 30 mL of toluene and collected
- N2 or roto-vap to NEAR dryness and proceed to 11.8.

11.7. Sample Dilution Procedure

11.7.1. Simple dilutions: Dilutions from 2X to 50X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

(Concentration of the original extract) x (amount of aliquot taken) x (volume of diluted extract) = final concentration of dilution.

Ex: 50X dilution of original 10 g/20 µL sample
(10 g/20 µL) x (2 µL aliquot + 98 µL keeper) = 1 g/100 µL FV

Record the final sample concentration on the extract label.

11.7.2. Complex dilution requiring respiking of IS and RS: Dilutions greater than 50x must be done by diluting and respiking the extract with IS and RS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IS and 20 µL RS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

11.8. Analytical Procedures

11.8.1. With a stream of dry, purified nitrogen, reduce the extract volume to approximately 100 µL. Add 20 µL of the recovery standard solution (Table 2). With a stream of dry, purified nitrogen, reduce the extract to volume to 20 µL.

11.8.1.1. Transfer the extract to an autoinjection vial and store in the dark at room temperature.

11.8.1.2. A smaller final volume can be used to decrease the detection limit upon client approval.

11.8.1.3. A larger final volume can be use to decrease potential matrix interferences of which cleanups were unsuccessful.

11.8.2. Inject a 1 or 2 µL aliquot of the extract into the GC, operated under the conditions previously used (Section 6.2) to produce acceptable results with the performance check solution.

11.8.3. Acquire SIM data according to Section 6.1.3. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Section 10). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is used to monitor their interference during the characterization of PCDFs.

11.8.4. Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

11.8.4.1. Retention Times

11.8.4.1.1. For 2,3,7,8-substituted congeners, which have an isotopically labeled internal or recovery standard present in the sample extract, the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled internal or recovery standard at m/z corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.

11.8.4.1.2. For 2,3,7,8-substituted compounds that do not have an isotopically labeled internal standard present in the sample extract, the relative retention time (relative to the appropriate internal standard) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to 13C-OCDD as determined from the daily routine calibration results.

11.8.4.1.3. For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

11.8.4.1.4. The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965

and 321.8936) must reach a maximum simultaneously (± 2 seconds).

11.8.4.1.5. The ion current responses for both ions used for the labeled standards (e.g., for 13C-TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously (± 2 seconds).

11.8.4.2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Table 9.

11.8.4.3. Signal-To-Noise Ratio

All ion current intensities must be >2.5 times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 6 describes the procedure to be followed for the determination of the S/N.

11.8.4.4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N >2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

12. DATA ANALYSIS AND CALCULATIONS

12.1. For gas chromatographic peaks that have met the criteria outlined in Section 11.8.4 calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times RRF(n)}$$

Where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,

Ax = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDD/PCDFs,

Ais = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standards,

Qis = quantity, in pg, of the internal standard added to the sample before extraction,

W = sample size in g (if solid) or L (if liquid).
RRF(n) = Calculated mean relative response factor for the analyte

[RRF(n) with n = 1 to 17; Section 10.3.5].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, RRF(n) is the value calculated using the equation in Section 10.3.5.1. However, if it is a non-2,3,7,8-substituted congener, the RRF(k) value is the one calculated using the equation in Section 10.3.5.3.2 [RRF(k) with k = 27 to 30].

12.2. Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{Internal Standard Percent Recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times RRF(m)} \times 100$$

Where:

Ais = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard,

Ars = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (see Table 5, footnotes),

Qis = Quantity, in pg, of the internal standard added to the sample before extraction,

Qrs = Quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

RRF(m) = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 5, footnotes) recovery standard. This represents the mean obtained in Section 10.3.5.4 [RRF(m) with m = 18 to 26].

12.3. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limit (MCL) for that compound listed in Table 1, the linear range of response versus concentration may have been exceeded. In such cases, the following corrective actions will be undertaken:

12.3.1. If the signal for the analyte has saturated the detector, a single dilution and reanalysis of the extract will be made in an attempt to bring the signal within the range of the detector. If the measured concentration of the analyte is still above the MCL, the reported concentration for the analyte will be qualified appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.3.2. If the signal for the analyte is above the MCL but does not saturate the detector, the concentration will be reported and qualified appropriately. Some

programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.4. In either case, **with the approval of the client**, the sample may be re-extracted and/or re-analyzed with one or more of the following adjustments made to the analytical procedure in order to provide a concentration which meets client-specific data quality objectives.

12.4.1. Extraction and analysis of a one tenth aliquot. This is appropriate if it will provide analyte concentration within the MCL and a representative sample aliquot.

12.4.2. Extraction of an aliquot large enough to be representative with an increased concentration of internal standard and surrogate spike components added prior to the extraction. The extract is then diluted either prior to or after the cleanup procedures.

12.4.3. Dilution of the original extract. Internal standard components are re-spiked at an appropriate level prior to analysis. In this case, the internal standard recoveries are taken from the original analysis.

12.5. For the other congeners (including OCDD and OCDF), however, report the measured concentration and indicate that the value exceeds the upper calibration standard.

12.6. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.

12.7. Sample-Specific Estimated Detection Limit

The sample-specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

12.7.1. Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

12.7.1.1. Use the expression for EDL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N <2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the internal standard (if the congener possesses an internal standard) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a 13C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their internal standard must be consistently paired (using either both lighter mass ions or both heavier mass ions).

Use the formula:

$$EDL_{Specific2,3,7,8-subst.PCDD/PCDF} = \frac{2.5 \times H_x \times Q_{is}}{H_{is} \times W \times RRF(n)}$$

$$\text{EDL(specific 2,3,7,8-subst. PCDD/PCDF)} = \frac{2.5 \text{ x } H_x \text{ x } Q_{is}}{H_{is} \text{ x W x RRF(n)}}$$

Where:

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs.

H_x = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

H_{is} = height of one of the quantitation ions (Table 6) for the labeled internal standards.

W, RRF (n), and Qis retain the same meanings as defined in Section 12.1

12.7.2. Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

12.7.2.1. When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 11.8.4, calculate the "Estimated Maximum Possible

Concentration" (EMPC) according to the expression shown in Section 12.1, except that Ax in Section 12.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDL can be calculated using the above formula and the height of one of the ions as appropriate.

12.8. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

S_1 and S_2 represent sample and duplicate sample results.

12.9. The 2,3,7,8-TCDD toxic equivalents (TEQ) of PCDDs and PCDFs present in the sample are calculated at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the seventeen 2,3,7,8-substituted PCDDs and PCDFs (Table 11). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 11.

12.9.1. Two-GC Column TEF Determination

12.9.1.1. The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be ≤ 25% valley.

12.9.1.2. For samples that have a presumptive positive result for 2,3,7,8-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2,3,7,8-TCDF is then the result calculated from the DB-225 analysis. The chromatographic separation between 2,3,7,8-TCDF and any other unlabeled TCDF isomers must be < 25% valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in Section 12.1 through 12.6.

12.9.1.3. A DB-225 column can be used in the quantitative analysis of 2,3,7,8-TCDF and 2,3,7,8-TCDD analytes. Since the DB-225 cannot resolve 2,3,7,8-TCDD any positively identified 2,3,7,8-

TCDD which exceeds the reporting limit shall be confirmed on a DB-5 column.

12.9.1.4. For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 11.5.4) and signal-to-noise ratio criteria. In addition, the retention time identification criterion described in Section 11.5.4 applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 12 and the results from the routine calibration run on the DB-5 column.

13. METHOD PERFORMANCE

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. Figure 3 provides a typical 12-hour analysis sequence. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

13.1. GC Column Performance

13.1.1. Inject 1 or 2 µL of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.3 within a total cycle time of < 1 second.

13.1.2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of ≤ 25 percent (Figure 4),

Where:

$$\text{Valley Percent} = (x/y) \times 100$$

x = measured as in Figure 4 from the 2,3,7,8-closest TCDD eluting isomer,

y = the peak height of 2,3,7,8-TCDD

13.1.3. It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for

2,3,7,8-TCDD must be labeled on the chromatograms. The chromatograms showing the first and last eluters of a homologous series must be included.

13.1.4. The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

14. POLLUTION PREVENTION

14.1. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.

14.2. The use of Roto-vaps and Turbo-vaps rather than Kuderna-Danish reduction allows extraction solvents to be collected and disposed of rather than released to the atmosphere.

14.3. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.4. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.

14.5. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

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

15.1. Extracted aqueous/leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Extracted soil samples and thimbles, extracted PUF filters, XAD-2 resin, paper funnel filters, glass wool, sodium sulfate, assorted disposable glassware, fish/crawfish or similar materials contaminated with methylene chloride. Silica gel, alumina, carbon and sodium sulfate, from column clean-ups, contaminated with various solvents and eluates. Dump the materials into a contaminated lab trash bucket. When the bucket is full or at

the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Flammable solvent waste generated during glassware and sodium sulfate cleaning. Flammable solvent waste collected during roto-vap/turbo-vap reduction of extracted samples. Keep waste flammable solvents separated from waste methylene chloride. Collect the waste flammable solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.4. Waste methylene chloride generated during quartz fiber filter preparation, PUF adsorbent preparation, XAD-2 resin preparation, PUF/XAD-2 cartridge preparation, glassware rinsing and sodium sulfate pre-rinsing.. Waste methylene chloride collected during roto-rap/turbo-vap reduction of extracted samples. Keep waste methylene chloride separated from waste flammable solvents. Collect the waste methylene chloride in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel methylene chloride drum in the H3 closet. When the drum is full to between one and four inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.5. Contaminated mercury used during extract cleanup. Pour the contaminated mercury into a 250-ml plastic bottle labeled for contaminated mercury. When full or after one year, whichever comes first, transfer this jar to the waste collection area for shipment.

15.6. Contaminated sulfuric acid used during extract cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

15.7. Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between one and four inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.

16. REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.3. Compendium Method TO-9A "Determination of Polychlorinated, Polybrominated, and Brominated, Cholorinated Dibenxo-p-dioxins and Dibenzofurans in Ambient Air", EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.4. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.5. "Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.6. "Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.7. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Deviations from reference method.

17.1.1. The method specifies that 2 µL injections are used throughout the analysis. If an instrument demonstrates adequate sensitivity and chromatographic resolution, then the analyst may use 1 µL injections for all performance checks, standards, QC samples, and samples.

17.1.2. In Section 2.7 of Method 8290, a retention time window of 0.005 RT units is used to tentatively identify unlabeled PCDD/PCDFs for which there are no corresponding labeled internal standards. All available labeled internal standards are used; therefore, a retention time window of -1 to +3 seconds is used to identify all compounds. See Section 7.8.4.1 of Method 8290.

17.1.3. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.1.4. Extract clean-ups are performed at the discretion of the analyst when

interferences are observed. Then, the analyst should select the clean-up procedure appropriate to the interferent.

17.1.5. Section 7.4.6.4 of Method 8290 indicates that extracts should be transferred with hexane, then toluene. Toluene is used to transfer extracts to maintain compound solubility and minimize analyte loss.

17.1.6. Section 7.5.1.2 of Method 8290 specifies that a NaCl solution should be used for partitioning. Instead, the laboratory uses laboratory water only. NaCl is used to break up emulsions that may form. An analyst may use NaCl, NaOH, or any other mechanical means to break up an emulsion.

17.1.7. Section 7.5.3 of Method 8290 specifies that hexane is used as a column elution solvent. The laboratory uses cyclohexane to achieve better and more reproducible separation of the target analyte from the interferent.

17.1.8. Carbon columns are packed with silica gel in place of celite. Elution solvents are changed accordingly. (SOP Section 11.4; Method 8290 Section 7.5.3.2).

17.1.9. Modifications from TO-9A method

17.1.9.1. Quartz Fiber Filters are cleaned by Soxhlet extraction with methylene chloride, not baked at 400 degrees C for 5 hours.

17.1.9.2. The PUF material may be pre-cleaned with methylene chloride or other appropriate solvent. The PUFs are not reused.

17.1.9.3. The 37CL-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.1.9.4. Samples are extracted with toluene not benzene.

17.1.9.5. Concentration is performed by rotary evaporation not Kuderna-Danish.

17.1.9.6. All cleanup procedures are optional and applied based on the analyst's discretion.

17.1.9.7. The laboratory uses 2 labeled recovery standard for the quantitation of labeled internal standards.

17.1.9.8. The final volume is adjusted to 20 µL in tetradecane.

17.1.9.9. Calibration and quantitation are performed in accordance to this SOP.

17.2. Summary of modifications to SOP from previous revisions

17.2.1. Modified to include extraction and analysis of ambient air samples collected in filter/PUF material.

17.3. Tables or figures referenced in body of SOP.

17.3.1. Table 1 - Types of Matrices

17.3.2. Table 2 - Composition of The Sample Fortification and Recovery Standard Solutions.

17.3.3. Table 3 - The Fifteen 2,3,7,8-Substituted PCDD and PCDF Congeners

17.3.4. Table 4 - Isomers of Chlorinated Dioxins and Furans

17.3.5. Table 5 - Concentrations of Calibration Solutions

17.3.6. Table 6 - Ions Monitored for PCDDs/PCDFs

17.3.7. Table 7 - Recommended GC Operating Conditions

17.3.8. Table 8 - Congeners in the GC Performance Evaluation Solution (DB-5)

17.3.9. Table 9 - Theoretical Ion Abundance Ratios and Control Limits

17.3.10. Table 10 - Relative Response Factor Attributes

17.3.11. Table 11 - 2,3,7,8-TCDD Equivalent Factors

17.3.12. Table 12 - TEF: Analyte Relative Retention Time Reference Attributes

17.3.13. Figure 1 - Analysis Flowchart

17.3.14. Figure 2 - Compound Structure

17.3.15. Figure 3 - Analysis Scheme

17.3.16. Figure 4 - GC Performance Check Chromatogram on the DB-5 Column

17.3.17. Figure 5 - PFK Peak Profile

17.3.18. Figure 6 - Manual Determination of Signal-to-Noise

17.3.19. Appendix A - Periodic Wipe Test Performance

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Human/ Fish Tissue	Adipose Tissue	Sludges, Fuel Oil	Still-Bottom	Ambient or Source Samples
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IS Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (μL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Sample Fortification and Recovery Standard Solutions

Analyte	Sample Fortification Solution Concentration pg/µL; Solvent: Isooctane	Recovery Standard Solution Concentration pg/µL; Solvent: Tetradecane
13C-2,3,7,8-TCDD	2[(a)], 100[(c)]	--
13C-2,3,7,8-TCDF	2[(a)], 100[(c)]	--
13C-1,2,3,4-TCDD	--	100
13C-1,2,3,7,8-PeCDD	2[(a)], 100[(c)]	--
13C-1,2,3,7,8-PeCDF	2[(a)], 100[(c)]	--
13C-1,2,3,6,7,8-HxCDD	2[(a)], 100[(c)]	--
13C-1,2,3,4,7,8-HxCDF[(d)]	2[(a)], 100[(c)]	--
13C-1,2,3,7,8,9-HxCDD	--	100
37Cl-2,3,7,8-TCDD[(b)(c)]	0.8[(b)],100[(c)]	
	100[(c)]	
13C-2,3,4,7,8-PeCDF[(c)]	100[(c)]	
13C-1,2,3,6,7,8-HxCDF[(c)(d)]	100[(c)]	
13C-1,2,3,4,7,8-HxCDD[(c)]	100[(c)]	
13C-1,2,3,4,7,8,9-HpCDD[(c)]	100[(c)]	
13C-1,2,3,4,6,7,8-HpCDD	2[(a)], 100[(c)]	--
13C-1,2,3,4,6,7,8-HpCDF	2[(a)], 100[(c)]	--
13C-OCDD	4[(a)], 200[(c)]	--

(a) Standard 8290, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) 13C-1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and 13C-1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 23 and Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The 13C-labeled analog is used as an internal standard.
(+)The 13C-labeled analog is used as a recovery standard.

TABLE 4

Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms

# of Chlorine Atoms	# of Dioxin Isomers	# of 2,3,7,8 Isomers	# of Furan Isomers	# of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	**75**	**7**	**135**	**10**

TABLE 5

High Resolution Concentration Calibration Solutions

Compound	Concentration (ng/mL)				
	CS1	CS2	CS3 (VER(6))	CS4	CS5
Native CDDs and CDFs					
2,3,7,8-TCDD	0.5	2	10	40	200
2,3,7,8-TCDF	0.5	2	10	40	200
1,2,3,7,8-PeCDD	2.5	10	50	200	1000
1,2,3,7,8-PeCDF	2.5	10	50	200	1000
2,3,4,7,8-PeCDF	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
OCDD	5.0	20	100	400	2000
OCDF	5.0	20	100	400	2000
Labeled CDDs and CDFs					
$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100
$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100

Compound	**Concentration (ng/mL)**				
	CS1	**CS2**	**CS3 (VER(6))**	**CS4**	**CS5**
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	100	100	100	100
$^{13}C_{12}$-OCDD	200	200	200	200	200
Cleanup Standard/ FS					
$^{37}Cl_4$-2,3,7,8-TCDD	0.5	2	10	40	200
Recovery Standards					
$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

TABLE 6*

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate[a] Mass	Ion ID	Elemental Composition	Analyte
1	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF (S)
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF (S)
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD (S)
	333.9338	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD (S)
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	[354.9792]	LOCK	C_9F_{13}	PFK
2	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF (S)
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF (S)
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	367.8949	M+2	$^{13}C_{12}H3^{35}Cl_4{}^{37}ClO_2$	PeCDD (S)
	369.8919	M+4	$^{13}C_{12}H3^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD (S)
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
	[354.9792]	LOCK	C_9F_{13}	PFK
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF (S)
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF (S)
	389.8156	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD (S)
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD (S)

TABLE 6 (cont.)*

Ions Monitored for HRGC/HRMS Analysis of PCDDs/PCDFs

Descriptor	Accurate[a] Mass	Ion ID	Elemental Composition	Analyte
4	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7788	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8250	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF (S)
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	423.7767	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD (S)
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}CL_2O_2$	HpCDD (S)
	479.7165	M+4	$C_{12}H^{35}CL_7{}^{37}Cl_2O$	NCDPE
	[430.9728]	LOCK	C_9F_{17}	PFK
5	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7780	M+2	$^{13}C1_2{}^{35}Cl_7{}^{37}ClO_2$	OCDD (S)
	471.7750	M+4	$^{13}C1_2{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD (S)
	513.6775	M+4	$^{13}C1_2{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE
	[442.9728]	LOCK	$C_{10}F_{17}$	PFK

[a] The following nuclidic masses were used:

H =	1.007825	O =	15.994915
C =	12.000000	^{35}Cl =	34.968853
^{13}C =	13.003355	^{37}Cl =	36.965903
F =	18.9984		

S = Internal/recovery standard

*The homologous groups for functions 1-3 do not use the same lockmass as described in Table 6. They use masses 316.9824, 366.9792, and 380.9760, respectively.

TABLE 7

Recommended GC Operating Conditions

The GC Operating Conditions (Temperatures (°C), and Times (minutes)) Are as Follows:

Injector Temperature: 280°C

Interface Temperature: 280°C

Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last of the tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

TABLE 8

PCDD and PCDF Congeners Present in the GC Performance Evaluation Solution and Used for Defining the Homologous GC Retention Time Windows on a 60-M DB-5 Column[(b)]

# of Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	Early Eluter	Late Eluter	Early Eluter	Late Eluter
4[(a)]	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,3,4,6,8	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,8	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

[(a)] In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, $^{13}C_{12}$-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present.

(b) The PCDF Congeners present in GC the Performance Evaluation Solution for the 30 m DB-225 column include:

- 1,2,3,9-TCDF
- 2,3,7,8-TCDF
- 2,3,4,7-TCDF
- $^{13}C_{12}$-2,3,7,8-TCDF

Column performance criteria is met when the percent valleys between the 2,3,7,8-TCDF analyte and the closest eluting isomers are $\leq$ 25%.

TABLE 9

Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs

# of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M / M+2	0.77	0.65	0.89
5	M+2 / M+4	1.55	1.32	1.78
6	M+2 / M+4	1.24	1.05	1.43
6[a]	M / M+2	0.51	0.43	0.59
7[b]	M / M+2	0.44	0.37	0.51
7	M+2 / M+4	1.04	0.88	1.20
8	M+2 / M+4	0.89	0.76	1.02

[a] Used only for ^{13}C-HxCDF (IS)
[b] Used only for ^{13}C-HpCDF (IS)

TABLE 10

Relative Response Factor [RRF (number)] Attributes

Number	Specific Congener Name
1	2,3,7,8-TCDD (and total TCDDs)
2	2,3,7,8-TCDF (and total TCDFs)
3	1,2,3,7,8-PeCDD (and total PeCDDs)
4	1,2,3,7,8-PeCDF
5	2,3,4,7,8-PeCDF
6	1,2,3,4,7,8-HxCDD
7	1,2,3,6,7,8-HxCDD
8	1,2,3,7,8,9-HxCDD
9	1,2,3,4,7,8-HxCDF
10	1,2,3,6,7,8-HxCDF
11	1,2,3,7,8,9-HxCDF
12	2,3,4,6,7,8-HxCdf
13	1,2,3,4,6,7,8-HpCDD (and total HpCDDs)
14	1,2,3,4,6,7,8-HpCDF
15	1,2,3,4,7,8,9-HpCDF
16	OCDD
17	OCDF
18	$^{13}C_{12}$-2,3,7,8-TCDD
19	$^{13}C_{12}$-2,3,7,8-TCDF
20	$^{13}C_{12}$-1,2,3,7,8-PeCDD
21	$^{13}C_{12}$-1,2,3,7,8-PeCDF
22	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
23	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
24	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
25	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
26	$^{13}C_{12}$- OCDD
27	Total PeCDFs
28	Total HxCDFs
29	Total HxCDDs
30	Total HpCDFs

TABLE 11

2,3,7,8-TCDD Equivalent Factors (TEFs) for the Polychlorinated Dibenzodioxins and Dibenzofurans

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCdd	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	OCDD	0.001
8	2,3,6,7-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	OCDF	0.001

TABLE 12

Toxicity Equivalency Factor:
Analyte Relative Retention Time Reference Attributes

Analyte	Analyte RRT Reference (a)
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to $^{13}C_{12}$-1,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF

FIGURE 1

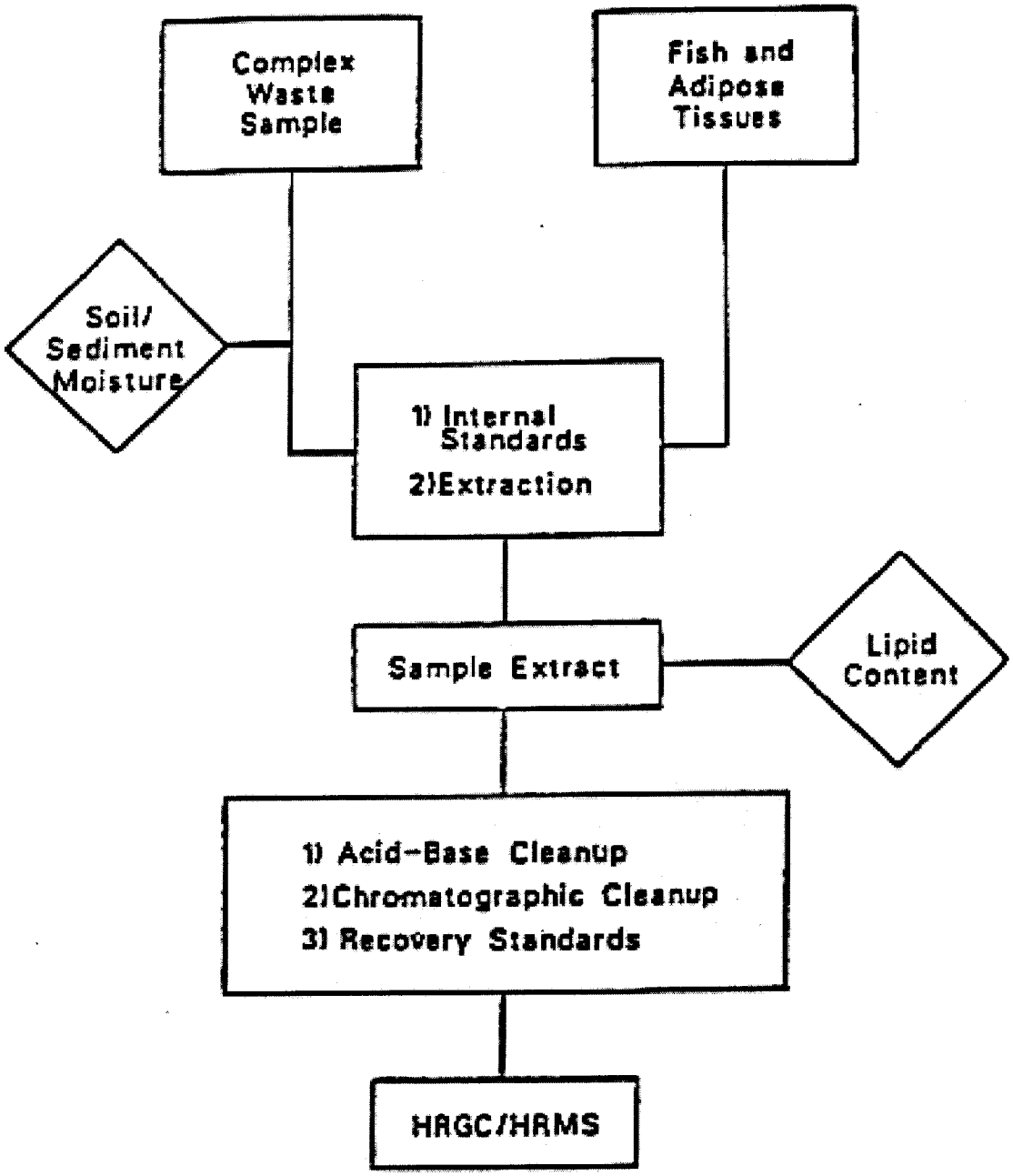
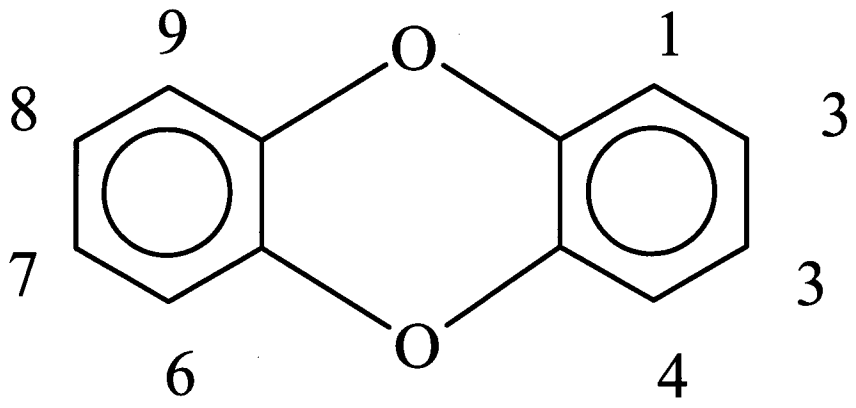
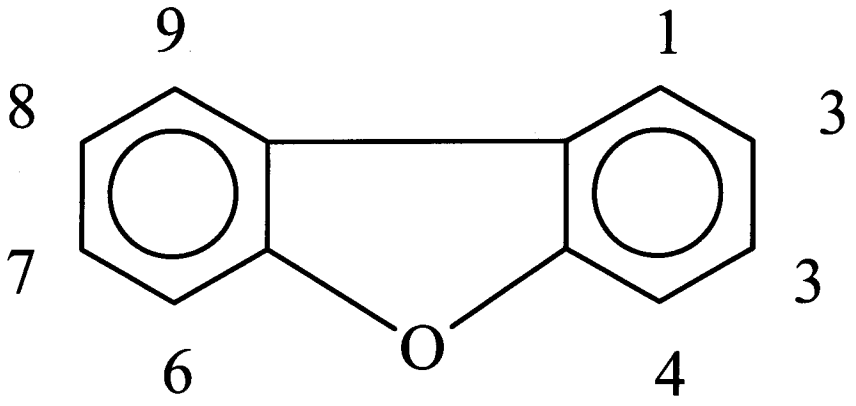

Complex Waste Sample
Fish and Adipose Tissues
Soil/ Sediment Moisture
1) Internal Standards
2) Extraction
Sample Extract
Lipid Content
1) Acid-Base Cleanup
2) Chromatographic Cleanup
3) Recovery Standards
HRGC/HRMS

FIGURE 2
Structure of Dibenzodioxin and Dibenzofuran



Dibenzodioxin



Dibenzofuran

FIGURE 3
Analytical Procedure

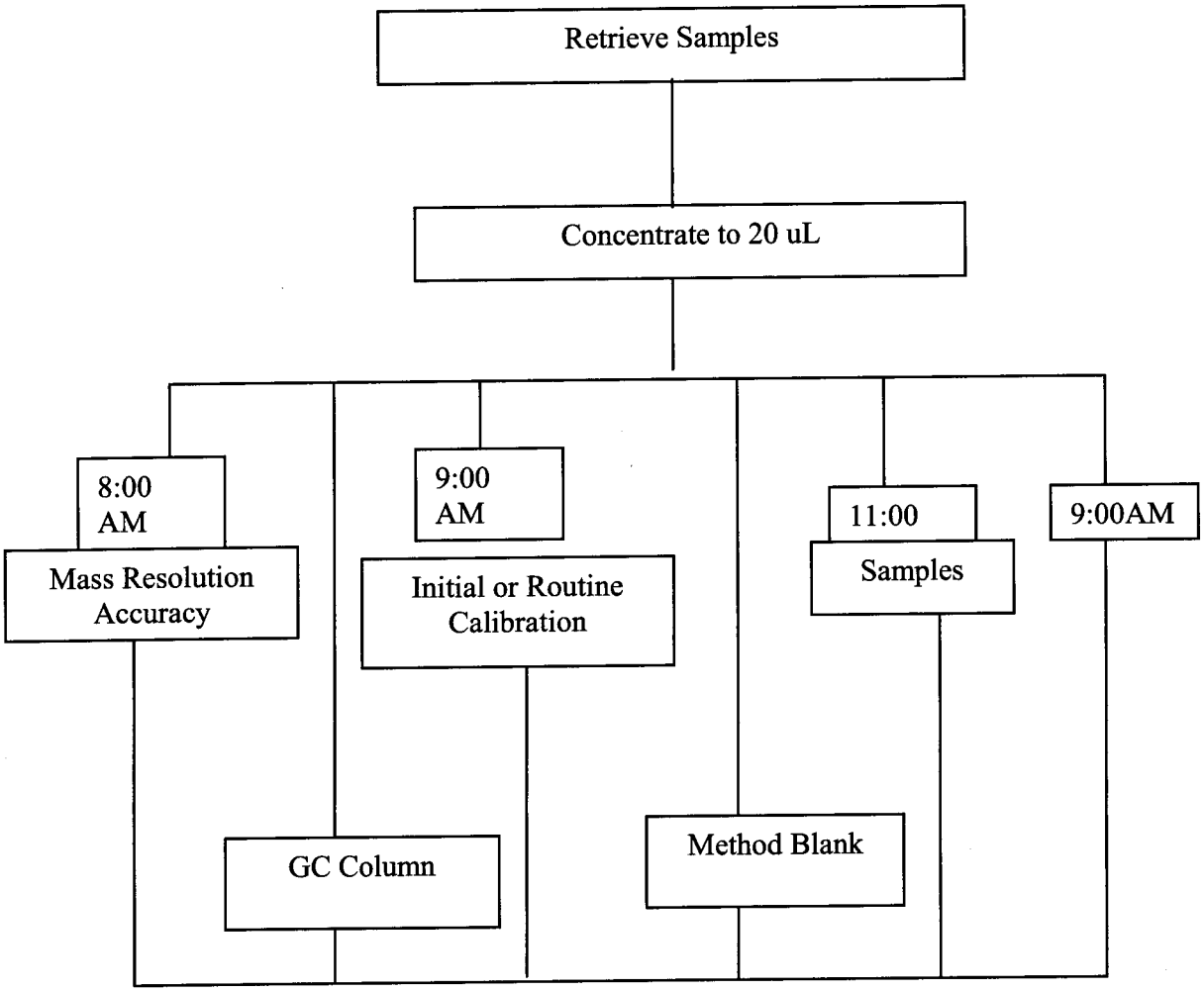


FIGURE 4

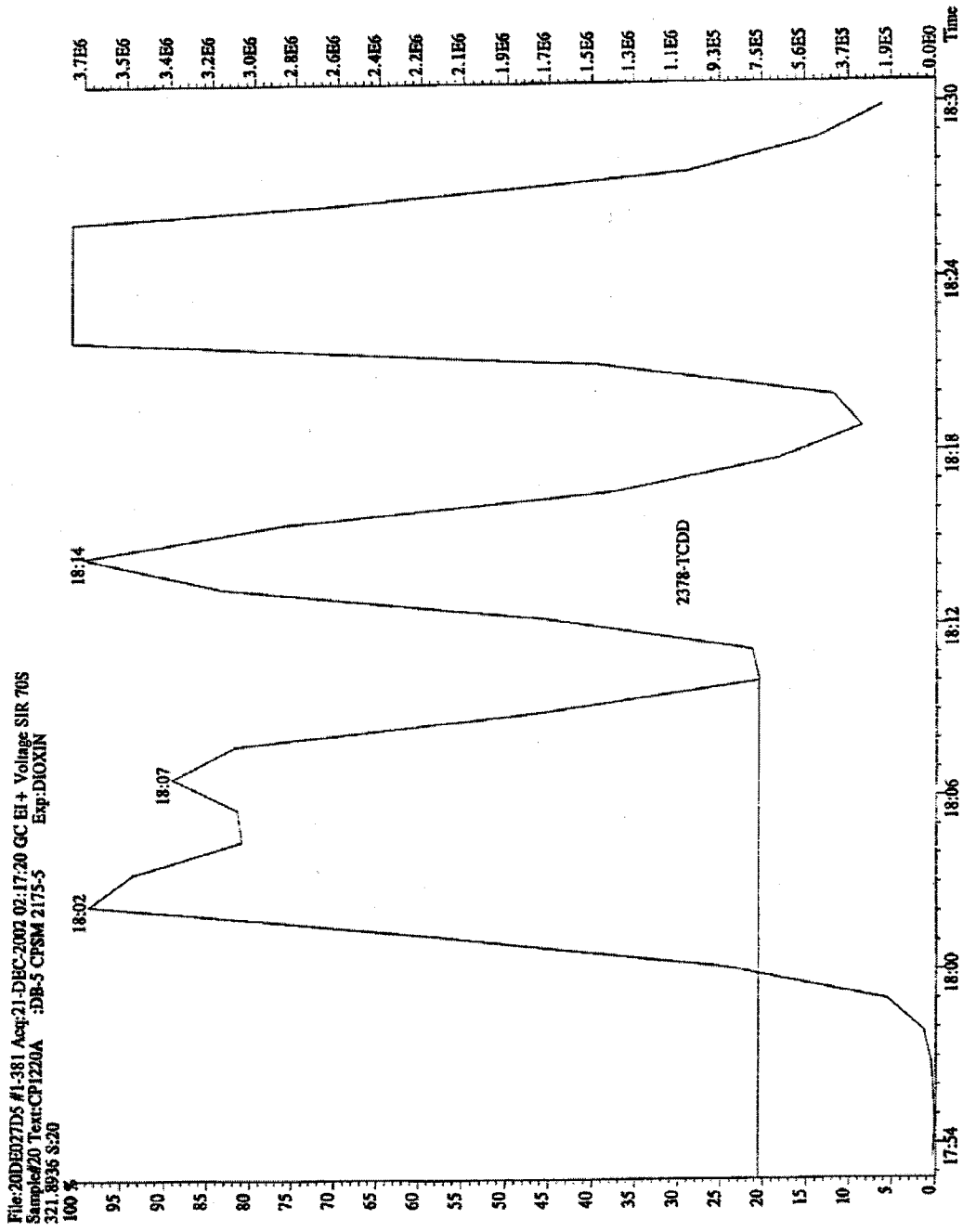

File:20DE027D5 #1-381 Acq:21-DEC-2002 02:17:20 GC EI+ Voltage SIR 70S
Sample#20 Text:CP1220A :DB-5 CPSM 2175-5 Exp:DIOXIN
321.8936 S:20
100 %
18:02
18:07
18:14
2378-TCDD
Time

Figure 5

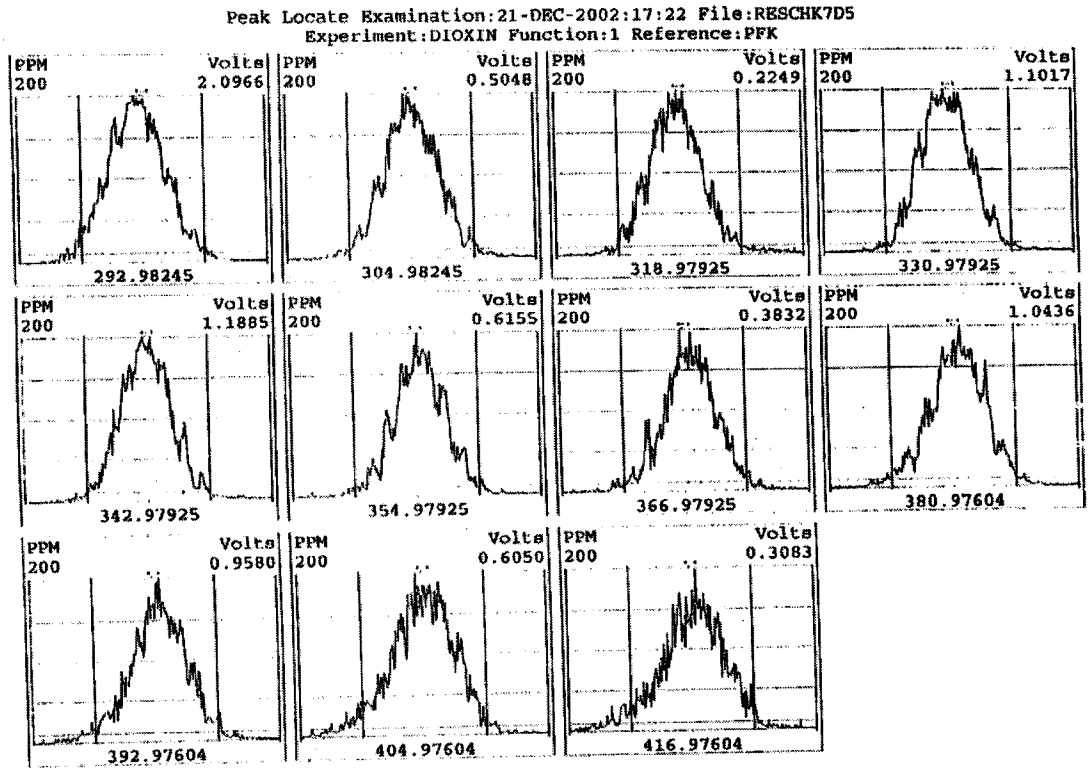
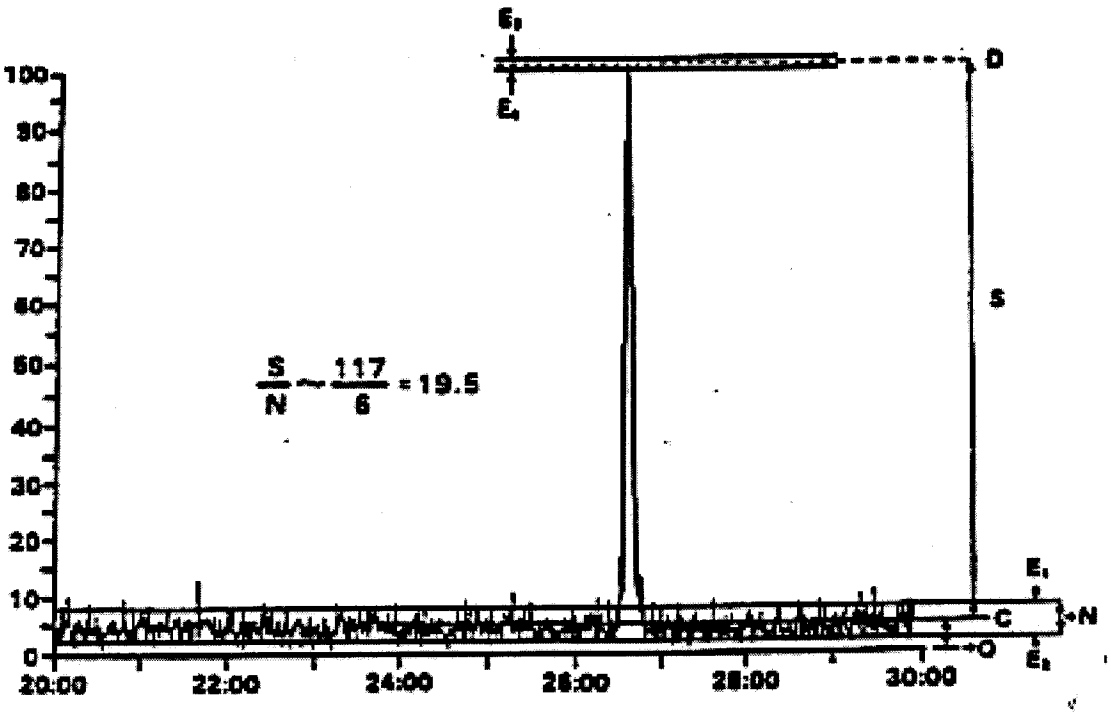

Peak Locate Examination:21-DEC-2002:17:22 File:RESCHK7D5
Experiment:DIOXIN Function:1 Reference:PFK
PPM 200 Volts 2.0966 292.98245
PPM 200 Volts 0.5048 304.98245
PPM 200 Volts 0.2249 318.97925
PPM 200 Volts 1.1017 330.97925
PPM 200 Volts 1.1885 342.97925
PPM 200 Volts 0.6155 354.97925
PPM 200 Volts 0.3832 366.97925
PPM 200 Volts 1.0436 380.97604
PPM 200 Volts 0.9580 392.97604
PPM 200 Volts 0.6050 404.97604
PPM 200 Volts 0.3083 416.97604

FIGURE 6



Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

APPENDIX A

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION

Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20 μL (either in a minivial or in a capillary tube). Inject 2 μL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS

Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

Title: TOXICITY CHARACTERISTIC LEACHING PROCEDURE AND SYNTHETIC PRECIPITATION LEACHING PROCEDURE

[SW846 Method 1311]

Approvals (Signature/Date):

Technology Specialist	3-18-08 Date	Health & Safety Coordinator	3/18/08 Date
Quality Assurance Manager	3/19/08 Date	Laboratory Director	3/20/08 Date
Technical Director	3/19/08 Date		

This SOP was previously identified as SOP CORP-IP-0004NC, Rev 1.2, dated 11/11/04

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1. SCOPE AND APPLICATION

1.1. This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW846 Method 1311. The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The other three are corrosivity, reactivity, and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions that were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.

1.2. The specific list of TC analytes and regulatory limits may be found in Appendix A.

Note: The list in Appendix A does not include the December 1994 EPA rule for Universal Treatment Standards for Land Disposal Restrictions. Those requirements include 216 specific metallic and organic compounds and, in some cases, lower detection limit requirements (see 40 CFR 268.40). TCLP leachates are part of the new Universal Treatment Standards, but the conventional analytical methods will not necessarily meet the new regulatory limits. Consult with the client and with TestAmerica Technical Specialists before establishing the instrumental methods for these regulations.

1.3. This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.

1.4. The procedure is applicable to liquid, solid, and multiphase wastes.

1.5. The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

1.6. The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

1.7. If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the procedure need not be run. If the total analysis results indicate that TCLP is not required, the decision to cease TCLP analysis should be remanded to the client.

1.8. If an analysis of any one of the liquid fractions of the procedure leachate indicates that a regulated compound is present at such a high concentration that, even after accounting for dilution from the other fractions of the leachate, the concentration would be equal to or above the regulatory level for that compound, then the waste is hazardous and it may not be necessary to analyze the remaining fractions of the leachate. However, the remaining analyses should not be terminated without the approval of the client.

1.9. Volatile organic analysis of the leachate obtained using a bottle extraction, normally used for extractable organics and metals, can be used to demonstrate that a waste is hazardous, but only the ZHE option can be used to demonstrate that the concentration of volatile organic compounds is below regulatory limits due to potential analyte loss into the headspace during the bottle extraction.

2. SUMMARY OF METHOD

2.1. For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP leachate.

2.2. For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis or recombination with the leachate. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater the extraction fluid employed is a pH 4.2 solution. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals, and/or, b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 µm fiber filter.

2.3. If compatible (i.e., multiple phases will not form on combination), the initial liquid filtrate of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3. DEFINITIONS

3.1. "Leachate" is used to refer to the solutions generated from these procedures (TCLP, SPLP, deionized water leach).

3.2. "Wet Solids" is that fraction of a waste sample from which no liquid may be forced out by pressure filtration.

4. INTERFERENCES

4.1. Oily wastes may present unusual filtration and drying problems. If requested by the client and as recommended by EPA (see Figure 3), oily wastes can be assumed to be 100% liquid and analysis for total concentrations of contaminants will be performed. This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter. Alternately, the oil may be subjected to pressure filtration. The portion that passes through the filter will be prepared and analyzed separately as an organic waste. The "wet solid" portion that remains behind on the filter will be subjected to leaching--prepared and analyzed. The results will then be mathematically combined.

4.2. Wastes containing free organic liquids (e.g., oil, paint thinner, fuel) usually require dilution prior to analysis to address the matrix interferences. In most instances this results in reporting limits elevated above the TCLP regulatory limits.

4.3. 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 as described in Section 9 and the individual determinative SOPs.

4.4. Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.

4.5. Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.

4.6. Overexposure of the sample to the environment will result in the loss of volatile components.

4.7. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized. CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.3. 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
Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Glacial Acetic Acid	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
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.

Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3-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.
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.
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.
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	2 mg/m^3-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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. A rotary agitation apparatus is used in this procedure. Certain samples may break the glass jars used in the procedure. For these samples, extra caution, including plastic or polyethylene overwraps of the glass jar, may be necessary. Turning the jar or bottle sideways rather than tumbling end over end may also reduce the chance of breakage. If sideways tumbling is used, note this change in the logbook comment section. Guards must be placed in front of any rotating equipment.

5.6. Secure tumbler and extraction apparatus before starting rotary agitation apparatus.

5.7. During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure.

5.8. Any cyanide containing waste or soil may result in the formation of hydrogen cyanide gas when exposed to acidic conditions. SPLP Fluid #3 reagent water must be used for these samples. **NOTE: Do not use an acidic SPLP fluid due to the potential release of hydrogen cyanide gas.**

5.9. Exposure to hazardous chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.10. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.11. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and Laboratory Supervisor.

5.12. Due to the potential for ignition, flammability or production of noxious fumes, do not attempt to dry non-aqueous liquid samples in an oven. Use extended drying in a ventilation hood.

6. EQUIPMENT AND SUPPLIES

6.1. Extraction vessels

6.1.1. For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Figure 2)

6.1.2. For metals - either borosilicate glass jars (2.5 L, with Teflon lid inserts) or 2.5 L HDPE (Nalgene or equivalent) bottles may be used

6.1.3. For non-volatile organics - only borosilicate glass may be used

6.2. Vacuum filtration apparatus and stainless steel pressure filtration apparatus (142 mm diameter), capable of 0 - 50 psi

6.3. Borosilicate glass fiber filters, 0.6 - 0.8 µm (Whatman GF/F 14.2 cm, 9.0 cm, 4.7 cm, 0.7 µm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use, or purchase pre-washed filters. Glass fiber filters are fragile and should be handled with care.

6.4. Rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Figure 1). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

6.5. ZHE Extract Collection Device: Gas-tight syringes, 50 or 100 mL capacity, Hamilton 0158330 or equivalent

6.6. Top loading balance, capable of 0 - 4000 ± 0.01g (all measurements are to be within ± 0.1 grams)

6.7. pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation

6.8. Magnetic stirrer/hotplate and stirring bars

6.9. VOA vials, 40 mL, with caps and septa

6.10. Glass bottles, 1 liter, with Teflon lid-inserts

6.11. Nalgene plastic bottles or equivalent, 1 liter

6.12. Miscellaneous laboratory glassware and equipment

7. REAGENTS AND STANDARDS

7.1. Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. For volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through activated carbon). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Hydrochloric acid, 1 N: Carefully add 83 mL concentrated reagent grade HCl to 800 mL reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.

7.3. Sodium hydroxide, 1 N: Carefully add 40 g reagent grade NaOH pellets to 800 mL reagent water, stir until the pellets are completely dissolved, cool and dilute to 1 liter with reagent water.

CAUTION: Heat is generated during this process.

7.4. Acetic acid, glacial: concentrated, reagent grade liquid (HOAc)

7.6. pH calibration solutions: buffered to a pH of 4, 7, and 10. Commercially available.

7.5. TCLP Leaching Fluids

7.5.1. General Comments

7.5.1.1. The pH of both solutions listed below should be monitored daily and the pH probes are to be calibrated prior to use.

7.5.1.2. The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.

7.5.1.3. Additional volumes of extraction fluids listed below may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.

7.5.1.4. At the end of the day, all remaining buffer solutions must be properly discarded.

7.5.2. TCLP Fluid #1: Carefully add 5.7 mL glacial acetic acid and 64.3 mL of 1 N NaOH to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. For 8 L of fluid use

45.6 mL glacial acetic acid and 514 mL 1N NaOH, dilute to 8 L with reagent water. When correctly prepared, the pH of this solution is 4.93 ± 0.05. The density of TCLP fluid #1 is 0.997 g/mL.

7.5.3. TCLP Fluid #2: Carefully add 5.7 mL glacial acetic acid to 500 mL reagent water in a 1 liter volumetric flask. Dilute to a final volume of 1 L with reagent water, cap and shake to mix well. For 8 L of fluid use 45.6 mL glacial acetic acid, dilute to 8 L with reagent water. When correctly prepared, the pH of this solution is 2.88 ± 0.05. The density of TCLP fluid #2 is 0.997 g/mL.

7.6. Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.

7.7. Sulfuric acid / nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

7.8. SPLP Leaching fluids

7.8.1. SPLP solutions are unbuffered and exact pH may not be attained. The pH of TCLP and SPLP fluids should be checked prior to use. If not within specifications, the fluid should be discarded and fresh fluid prepared.

7.8.2. SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 This fluid is used for soils from a site that is east of the Mississippi River and for wastes and waste waters.

7.8.3. SPLP fluid #2: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05. This fluid is used for soils from a site that is west of the Mississippi River.

7.8.4. SPLP fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

7.9. Methanol and methylene chloride - used to aid in cleaning oil contaminated equipment.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation. Undamaged glass containers should be washed according to SOP NC-QA-0014.

8.2. Samples being analyzed for metals only can be collected in either glass or polyethylene containers.

8.3. When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon lined septum capped vials with minimal headspace and stored at 4 ± 2°C). Samples should be opened only immediately prior to extraction.

8.4. Samples should be refrigerated to 4 ± 2°C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

8.5. The minimum TCLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than 50% solids), minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.

8.6. TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration (4 ± 2°C) until analyzed. ZHE leachates must be stored in VOA vials filled to eliminate all headspace.

8.7. Samples are subject to appropriate treatment within the following time periods.

Table 1
Holding Times (days)

Parameter	Collection to Start of TCLP Leach	End of TCLP Tumble to Preparation	Start of TCLP Leach or Semi- volatile Prep Extraction to Analysis	Total Elapsed Time
Volatiles	14	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Other Metals	180	N/A	180	360

NOTE: The initial holding time is measured from date of collection to date TCLP extraction started. (This should be the TCLP extraction date in QuantIms.) Semi-volatile method prep holding time is measured from the day tumbling is complete to the start of method extraction. Subsequent analysis holding times are measured from the date extraction (TCLP or method prep) starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit. The Total Elapsed Time is to be used as guidance. If preps are initiated at the last possible moment of a holding time, the elapsed times may be exceeded.

9. QUALITY CONTROL

9.1. Quality Control Batch (QC Batch) - QA-003 defines a QC Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same procedures, reagents and standards within the same time period. The same lot of reagents must be used within a batch. A minimum of one TCLP extraction blank (Method Blank), one Laboratory Control Sample (LCS), one Matrix Spike (MS), and one Matrix Spike Duplicate (MSD) will be prepared with each TCLP leachate batch.

9.2. TCLP Extraction Blanks - A minimum of one blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples extracted in a particular vessel type. The blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples). If particle size reduction was performed on any sample in the batch, an equipment blank will be generated by passing blank fluid through the particle reduction apparatus. ZHE Extraction vessels will be uniquely numbered. Consult the TestAmerica QC Program and the individual analysis SOPs for blank acceptance criteria.

9.3. Laboratory Control Sample (LCS) - A LCS is required with each batch of 20 or fewer samples. The LCS shall be generated after a batch of TCLP leachates have been

generated (i.e., at the time of the preparative digestion or extraction) by spiking an aliquot of the appropriate extraction fluid used for that batch or reagent water. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.4. Matrix Spike (MS/MSD) - Matrix spikes are used to monitor the performance of the analytical methods on the matrix and to assess the presence of interferences. A MS/MSD pair are required with each batch of 20 or fewer samples.

9.4.1. Matrix spikes are to be added after filtration of the TCLP leachate. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.

9.4.2. Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.5. Corrective Actions

9.5.1. Consult the TestAmerica QC Program and individual analysis SOPs for corrective action for blanks and LCS

9.5.2. Method of Standard Additions (MSA) shall be used for mercury if all of the following conditions are met:

- Recovery of the analyte in matrix spike is not at least 50%,
- The concentration of the analyte does not exceed the regulatory level, and
- The concentration of the analyte measured in the sample is within 20% of the appropriate regulatory level.

If the matrix spike recovery is 5% or less due to dilution or matrix interference, contact the project manager and client for guidance. The client should also be contacted prior to initiation of any MSA steps. Refer to the individual analysis SOPs for details on how to perform MSA analysis.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to appropriate analysis SOPs.

11. PROCEDURE

11.1. General Comments

11.1.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented on a Nonconformance Memo kept in the project file and described in the final report. The variation must be approved by a Project Manager, Technical Specialist, and QA Manager. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.1.2. All masses should be recorded to the nearest 0.1 g.

11.2. Preliminary Sample Evaluations (Refer to Flow Chart #1, Appendix D)

11.2.1. Determine the total volume of TCLP leachate (solid phase leachate plus liquid filtrate) that needs to be generated for analysis according to the following:

Table 2
Recommended TCLP Leachate Volume

Analysis	TCLP Required Volume (mL)	SPLP Required Volume (mL)
Volatiles	3 x 40	3 x 40
Semi-volatiles	500	1000
Pesticides	500	1000
Herbicides	500	1000
Metals	300	300

11.2.1.1. For TCLP and SPLP samples used for matrix spike and matrix spike duplicate analysis, two to three times the listed volumes are required.

11.2.2. Sample Description (determine sample matrix)

11.2.2.1. Solid - If the waste will obviously yield no free liquid when subjected to pressure filtration, then proceed to Section 11.2.5 or 11.4 (Bottle Extraction Procedure or ZHE Procedure).

11.2.2.2. Liquid - If the sample is a monophasic liquid, proceed to Section 11.2.3 (Percent Solid Determination).

11.2.2.3. Multiphasic – The sample has discernible layers (liquid/liquid or liquid/solid). If more than one container of multi-phasic materials is received from the field, each container might show different amounts of each phase. Consult client to determine sample selection alternatives (composite all sample containers, select one, resample, etc.) if this occurs.

11.2.3. Solids Determination

11.2.3.1. Determine Type of Filtration Apparatus and Process

11.2.3.1.1. Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent solids less than 5%. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 11.4 (ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with Section 11.2.3.2. The aliquot of sample used here cannot be used again for the ZHE extraction.

11.2.3.1.2. If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (expected to be < 0.5%) , vacuum filtration should be used initially. Proceed to determination of percent dry solids (Section 11.2.3.2)

11.2.3.1.3. If the sample is viscous (sludge, oil, or is expected to have solids content > 0.5%), use pressure filtration. Proceed to determination of wet solids (Section 11.2.3.3).

11.2.3.2. Determination of percent dry solids

11.2.3.2.1. Measure and record the weight of the filter. Load the filter into the filter holder and assemble vacuum filter apparatus.

11.2.3.2.2. Homogenize the waste, then transfer 100 g subsample to a glass beaker. Record the sample weight in the percent dry solids section of the logbook.

11.2.3.2.3. Turn on vacuum source. Transfer the sample to the vacuum filtration device attempting to spread the waste sample evenly over the surface of the filter. Be sure to transfer all

particulates from the beaker to the filter. Use a reagent water rinse if necessary.

11.2.3.2.4. Once all liquid has been pulled though the filter, remove the filter with the wet solids from the vacuum filtration apparatus.

11.2.3.2.5. Dry the filter and solid phase at 100 ± 20 ° C for approximately 15 minutes.

11.2.3.2.6. Remove the filter from the oven, and allow to cool in a desiccator.

11.2.3.2.7. Weigh and record the dry weight of filter + particulates.

11.2.3.2.8. Calculate and record the percent dry solids.

11.2.3.2.9. If the percent dry solids is ≥ 0.5%, repeat the drying step. Weigh and record the second filter + particulates dry weight. If the two weighings do not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.

11.2.3.2.10. If the dry solids result is ≥ 0.5%, proceed to Section 11.2.3.3 using a fresh wet portion of the multiphase waste.

11.2.3.2.11. If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary. Filter sufficient sample with either the pressure filtration system or ZHE system as described in Sections 11.3 and 11.4. The filtrate is the TCLP leachate.

11.2.3.3. Determination of wet solids

11.2.3.3.1. Assemble the pressure filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 µm filter membrane).

11.2.3.3.2. Homogenize the waste, transfer a minimum of a 100 mL subsample to the glass beaker. Measure and record the gross weight (logbook Column A).

11.2.3.3.3. Measure and record the tare weight of the filtrate collection bottle (logbook Column D).

11.2.3.3.4. Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty glass beaker and any residual sample (logbook Column B).

11.2.3.3.5. Calculate and record the net weight of sample used for testing (logbook Column C).

11.2.3.3.6. Slowly apply gentle pressure of 10 psi to the filtration apparatus. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter during a two-minute period.

11.2.3.3.7. If necessary, repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a two-minute period.

Note: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils, or any wastes which do not pass through the filter, are classified by the method as a solid.

11.2.3.3.8. Remove the filtrate collection bottle, weigh and record the gross weight (logbook Column E).

11.2.3.3.9. Calculate and record the net weight of filtrate (logbook Column F). This result will be used in the percent solids calculation.

11.2.3.3.10. To determine the amount of filtrate, place the exact same type and size container as the filtrate container next to the filtrate. Add water to the exact level as the filtrate container to the empty container. Transfer the water to a graduated cylinder and record the volume. This step will reduce the amount of contamination, which may exist from transferring the filtrate to a graduated cylinder.

11.2.3.3.11. Retain the filtrate for possible recombination with the leachate in Section 11.3.7. Retain the filter and wet solids for the leaching in Section 11.3.

11.2.3.3.12. For multiphase sample preparations, calculate the total weight of wet solids and record the result in logbook Column G.

11.2.4. Particle-size Reduction for Fluid Selection

11.2.4.1. The subsample used for fluid selection must consist of particles less than approximately 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

11.2.4.2. Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram.

11.2.4.3. If the sample contains particles greater than approximately 1 mm in diameter, crush, cut, or grind the solids to the required size.

11.2.4.4. Consult a supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick).

11.2.5. Determination of Appropriate Extraction Fluid

11.2.5.1. If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.

11.2.5.2. Follow times, temperature, and particle size specified in this section as closely as possible. If reaction time between the acid solution and solid waste is too short or too long, the procedure may produce false pH readings.

11.2.5.3. For SPLP, refer to Section 7.8 for fluid selection. Record the fluid type in the logbook.

11.2.5.4. The TCLP leaching fluid for all volatiles is TCLP Fluid #1.

11.2.5.5. TCLP leach fluid determination for non-volatile analytes

11.2.5.5.1. Calibrate the pH meter with fresh buffer solution in accordance with the pH SOP.

11.2.5.5.2. Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a glass or plastic container, and record in the logbook. Note: If sample quantity is limited, consult supervisor or manager.

Note: Many multiphase samples have limited solids quantity . In these instances, use a 5 g aliquot of the whole sample. Document this difference in the logbook comment section.

11.2.5.5.3. Add 96.5 ± 1.0 mL of reagent water, add magnetic stir bar, cover with a watchglass, and stir for 5 minutes.

11.2.5.5.4. Measure and record the pre-test sample pH in the logbook.

Note: To avoid damaging a glass pH probe when organic liquid is present, use narrow range pH indicator paper or an ISFET pH meter.

11.2.5.5.5. If the pH is less than or equal to 5.0, use TCLP Fluid #1.

11.2.5.5.6. If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl. Slurry the sample briefly. Insert therm into room temp DI water in one vial in each pre-test sample group to monitor the temperature. All samples in the group must be heated at the same time in order for the temperature of the one monitored sample to represent the others. Heat to 50 ± 2°C and maintain for ten minutes.

Note: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

11.2.5.5.7. Cool to room temperature.

11.2.5.5.8. Measure and record the pH immediately after the sample has reached room temperature.

11.2.5.5.8.1. If the pH is less than or equal to 5.0, use TCLP Fluid #1. Record the buffer in the logbook.

11.2.5.5.8.2. If the pH is greater than 5.0, use TCLP Fluid #2. Record the buffer in the logbook.

11.2.6. For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 11.3.

11.2.7. For samples requiring analysis for volatile organics (ZHE), proceed to Section 11.4.

11.3. Bottle Extraction Procedure: Non-Volatile Constituents: Semi-Volatiles, Pesticides, Herbicides, Metals (Refer to Flow Chart #2, Appendix D)

11.3.1. Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram. If particle size reduction was required, record this in comments column in logbook.

11.3.1.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Determination of particle size reduction tools should take into account the requested analytes (e.g. avoid chromium steel tools when TCLP metals have been requested). Bricks, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction (contact PA or PM). Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

11.3.2. Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 11.2.1.

11.3.3. Use 100 g of solid unless sample quantity is limited. If limited sample, divide the total volume of solid phase leachate required by 20 to determine the minimum mass of solid phase required for leaching. Round this mass UP to the nearest 5g. Client must be notified if less than 100 g of solid material is used.

Note: Solid phase material is often in limited quantity from multiphase samples. Generally all the *solid* phase material and the filter from Section 11.2.3.3.11 are transferred to the leaching bottle.

11.3.4. All non-ZHE extraction vessels should be uniquely numbered. If breakage occurs, replacement vessels must be numbered with the original vessel identification number prior to use. Breakage must be noted in the logbook comments identifying the affected sample and vessel number.

11.3.5. Weigh the required mass of solid phase into an appropriate extraction vessel (plastic for metals only, glass for all others) and **slowly** add 20 times its mass of appropriate leaching fluid (e.g., 100 g of sample would require 2000 mL of leaching fluid). Record the weight of the sample aliquoted for the extraction. Record the volume of extraction fluid added in the logbook if other than 2000 mL. Record the pH.

11.3.6. Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room should be 23 ± 2°C. Record the rotary agitator I.D. and the date and time extraction is started and completed in the logbook.

Note: As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.

11.3.7. After tumbling in the rotary agitator is completed, remove the bottle and allow the solids to settle. Record the date and time the extraction is completed in the logbook. If sample was multiphase with an initial filtrate, drop a few drops of the filtrate (with a disposable glass pipette) into the extraction bottle and observe whether the filtrate is insoluble or forms a precipitate with the leachate. If so then the filtrate is not compatible with the leachate and must be bottled and analyzed separately. The results are normally mathematically recombined (Section 12.1.2). If the filtrate is compatible with the leachate (i.e., completely soluble) then pour the entire filtrate into the leachate bottle, recap and mix. Proceed with the leachate filtration step in the next section.

11.3.8. Filter the sample using pressure filtration by filtering through a new glass fiber filter. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered; however, sufficient volume should be generated to support the required analyses.

11.3.9. Measure the pH of the TCLP leachate and record in the logbook. (Use narrow range pH paper or ISFET pH meter to measure the pH of oily samples as a glass pH probe may be damaged.)

11.3.10. Prepare sufficient volume for MS/MSD quality control testing. Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

11.3.11. Immediately preserve the leachate as follows:

Metals	pH < 2 w/ HNO_3 for aqueous filtrates and leachates (do not acidify oils and other non-aqueous liquids)
All others	Refrigerate to 4 ± 2 °C

Note: Refer to Section 8.6 if precipitation occurs upon preservation.

11.3.12. Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis. For multiphase samples requiring mathematical recombination provide copies of the TCLP preparation logbook sheets to the sample preparation and analysis groups. Most mathematically recombined samples will require data entry for the filtrate and leachate portions as well as for the mathematically recombined results. Contact the Project Manager to ensure the proper sample login is completed.

11.4. ZHE Extraction Procedure: Volatile Constituents (Refer to Flow Chart #3, Appendix D)

11.4.1. Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, herbicides and semi-volatile organics).

11.4.2. Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere any longer than necessary.

11.4.3. Install new O-rings and adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

11.4.4. If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size as measured with a ruler (Do NOT sieve the sample). Size

reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram If particle size reduction was required record this in the comments column of the logbook.

Note: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4°C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate. Also see Section 11.3.1.

11.4.4.1. Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick). Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Bricks, rocks, or other solids amenable to grinding may be subcontracted out for particle size reduction (contact PM).

11.4.5. Homogenize and transfer an appropriate size subsample of the waste into the ZHE and record the mass in the logbook.

11.4.5.1. For wastes that are solid, a 25 g sample is used.

11.4.5.2. For wastes containing < 0.5% solids, the liquid portion of the waste, after filtration, is defined as the TCLP leachate. Filter enough of the sample to support all of the volatile analyses required.

11.4.5.3. If the sample has ≥ 0.5% solids and has non-volatile TCLP/SPLP requested, the appropriate sample size should be estimated based on the wet solids content determined in Section 11.2.3.3. If ZHE only, use visual wet solids estimate to sample subaliquot.

Note: For wastes containing greater than 0.5% wet or dry solids, the "solids" value from the ZHE filtration process may be used to determine the volume of fluid to load into the ZHE. This approach is recommended since the solids value from Section 11.2.3.3 may differ from the ZHE filtration solids due to sample variability or differences in the filtration apparatus.

11.4.6. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

11.4.7. Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

11.4.8. If the waste is solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 11.4.13.

11.4.9. If this is a mutliphase sample, carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.

11.4.10. Assemble a syringe and place the plunger in all the way. Attach the pre-weighed syringe to the liquid inlet/outlet valve and open the valve. Record the tare weight of the collection syringe in Column D of the logbook. .

11.4.11. Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a two-minute period.

Note: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe, weigh, record weight in Column E and filtrate volume in the logbook. Return to Section 11.4.10.

11.4.12. Repeat previous step increasing the pressure in 10 PSI increments until 50 psi is reached and no significant liquid has passed in a 2 minute period. Close the valve and discontinue gas pressure. Remove the collection device and record the total weight of the collection device with filtrate in column E and filtrate volume in the logbook. Transfer the filtrate to VOA vials and label appropriately. Calculate the weight of filtrate collected and record in Column F in the logbook.

Note: If the original waste contained less than 0.5% solids (Section 11.2.3.2), this filtrate is defined as the TCLP leachate and you may proceed to Section 11.4.22. Otherwise, save the vials by storing at 4°C under minimal headspace conditions, for recombination as in Section 11.4.21.

The material remaining in the ZHE is defined to be the "solid" phase. Calculate the weight of the solid phase and record in Column G of the logbook by subtracting the weight of the filtrate from the weight of the sample.

11.4.13. Determine the amount of buffer to use. Solid samples use 500 mL of leach fluid (20 X 25 g). For multiphase samples use the wet solids (Column G) amount and multiply by 20. Record the leach fluid volume in Column H of the logbook.

Note: The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only SPLP fluid #3.

11.4.14. Load the fluid transfer reservoir with an excess of Fluid #1 and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.

11.4.15. Attach the transfer line to the liquid inlet/outlet valve and open the valve. Carefully pump the required volume into the ZHE and close the valve. Disconnect the transfer line.

11.4.16. Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) two or three times. Reposition the ZHE in the vertical position.

11.4.17. Pressurize the ZHE to 5-10 psi. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the analysis.

11.4.18. Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.

11.4.19. Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Room temperature should be 23 ± 2 °C. The room temperature is recorded using a continuous temperature monitor.

11.4.20. Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was NOT maintained, return to Section 11.4 and repeat the leachate with a new aliquot of sample.

11.4.21. If there is an initial liquid filtrate (Sec 11.4.12) determine if it is compatible with the leachate if the filtrate has not been previously tested (Sec. 11.3.7).

11.4.21.1. Remove the plunger from the syringe and attach the barrel to the ZHE vessel. Open the outlet valve and pressurize as necessary to transfer about 1 mL of leachate into the syringe. Close the outlet valve.

11.4.21.2. With a glass pipette transfer a few drops of initial filtrate into the open syringe barrel. Formation of separate layers or a precipitate indicates the filtrate and leachate are not compatible. Bottle the filtrate for separate preparation and analysis. The results are normally mathematically recombined.

11.4.21.3. If the filtrate is compatible gently pour the remainder of the filtrate into the syringe barrel. Install the plunger. Bleed any pressure in the

ZHE piston. Open the inlet/outlet valve and depress the syringe plunger to inject the filtrate into the ZHE vessel. Do not inject the air bubble (if present) from the syringe.

11.4.21.4. Close the valve and rotate a few times to mix. Proceed with leachate filtration as described in the next section.

11.4.22. Attach an empty syringe to the outlet valve. Open the valve and pressurize the piston to expel the leachate from the ZHE vessel. Following collection, store the TCLP leachate in 2 or 3 40-mL VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic analysis procedure (see Section 16.3).

11.4.23. If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.2. Provide copies of the TCLP preparation logbook sheets to the sample preparation and analysis groups. Most mathematically recombined samples will require data entry for the filtrate and leachate portions as well as for the mathematically recombined results. Contact the project manager to ensure the proper sample login is completed.

11.4.24. ZHE Vessel Cleaning

11.4.24.1. Disassemble the vessel.

11.4.24.2. Clean all parts (vessel, lid, bottom, piston, and metal filters) with soapy water.

11.4.24.3. Rinse all parts with tap water followed by D.I. water.

11.4.24.4. Discard all used gaskets.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculations

12.1.1. Calculation of weight of extraction fluid to use:

Volume of extraction fluid = 20 X weight of wet solids to be extracted

12.1.2. Mathematical recombination of analytical results:

$$Final\ Analyte\ Concentration = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

Where,

V_1 = total volume of the initial filtrate phase (L).
C_1 = analyte concentration in initial filtrate phase (mg/L).
V_2 = volume of the theoretical solid phase leachate (L).
C_2 = analyte concentration in solid phase leachate (mg/L).,

12.2. Reporting Requirements

12.2.1. Follow these reporting conventions for multi-phase samples.

12.2.1.1. If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

12.2.1.2. If both phases are "ND" (not detected) the recombined result is "ND," and the reporting limit is calculated from the reporting limit for each phase.

12.2.1.3. If one phase is "ND" and the other phase has a positive result, use the zero for the "ND" phase and the positive value for the other phase to calculate the combined result. This will produce a minimum known concentration. Alternatively, at client request, the maximum possible concentration can be calculated by using the reporting limit for the "ND" phase rather than zero. The combined reporting limit is based on the reporting limit for both phases

12.2.2. Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

12.2.3. For limits and significant figures, consult the appropriate analytical methods (Section 16.3).

12.2.4. Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet or an NCM form. Some examples of such anomalies are:

12.2.4.1. Sample was monolithic - particle size reduction not possible due to nature of matrix.

12.2.4.2. Insufficient sample - less than the required 100 g minimum was available.

12.3. Review Requirements

12.3.1. Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation was properly documented in an NCM.

12.3.2. If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$Total \geq 20 \times TCLP$$

Note: Assumes the sample is 100% Solids.

12.3.3. Total constituent analysis results can be used to demonstrate the TCLP protocol is unnecessary. In performing a TCLP analysis, there is a 20:1 dilution of the original sample with the leaching solution. Thus, if the "total constituent" result is less than 20 times the TC level, it is impossible for the leachate to "fail" and the TCLP does not need to be performed. For example, the TC level for lead is 5.0 mg/L (ppm). Therefore, if a sample of lead-contaminated soil contains less than 100 ppm total lead, a TCLP test need not be run to demonstrate that lead is less than the TCLP limit.

13. METHOD PERFORMANCE

13.1. Refer to individual analysis SOPs.

13.2. Training Qualification:

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the

potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

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

15.2.1. Acidic waste from sample extract. This waste is collected in the laboratory in a designated container identified as "Acid Waste".

15.2.2. Buffer solutions. This waste can be poured down the drain with copious amounts of water.

15.2.3. Solid waste from sample extract, solid sample waste and used filter paper from the sample filtration step. This waste is disposed of in a designated container identified as "Solid Waste."

15.2.4. Flammable solvent waste and remaining TCLP extracts. This waste is disposed of in a flammable liquid solvent container identified as "Mixed Flammable Solvent Waste".

15.2.5. Glassware contaminated with acidic sample residue. Broken or unusable glassware is disposed of in a designated container identified as "Solid Waste." Glassware used in this test method is washed per the Glassware Washing SOP.

16. REFERENCES

16.1. Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.

16.2. Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1994, SW-846 Update II.

16.3. Related Documents

16.3.1. Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990

16.3.2. Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990

16.3.3. Technical Background Document and Response to Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April 1989

16.3.4. SOP QA-003, Quality Control Program

16.3.5. SOP CORP-IP-0003NC: Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods

16.3.6. SOP NC-MT-012: Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, Method 6010B and Method 200.7

16.3.7. SOP CORP-MT-0005NC: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1

16.3.8. SOP CORP-MS-0002NC: Determination of Volatile Organics by GC/MS based on Methods 8260B

16.3.9. SOP CORP-MS-0001NC: GC/MS Analysis Based on Method 8270C

16.3.10. SOP NC-GC-038: Gas Chromatographic Analysis Based on Methods 8000B, 8021B, 8081A, 8082, 8151A, and 8015B

16.3.11. NC-OP-0031: Extraction Procedure for Chloriantaed Acid Herbicides based on Method 8151A

16.3.12. SOP NC-OP-032: Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, and 600 Series Methods

16.3.13. SOP NC-QA-0017: Standards and Reagents

16.3.14. SOP NC-WC-0010: pH Electrometric Method

16.3.15. SOP NC-QA-0014: Glassware Washing

17. MISCELLANEOUS

17.1. Modifications/Interpretations from Reference Methods

17.1.1. Section 11.2: Preliminary Evaluations. Section 7.1 of the source Method 1311 states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., <

100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples which have been subjected to the oven-drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

17.1.2. Sections 11.3.7 and 11.4.21: Determination of Filtrate/Extraction Fluid Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed.

17.1.3. Section 9.2: TCLP Extraction Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." TestAmerica has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used.

17.1.4. Section 11.2.5.5.8.1: Determination of Appropriate Extraction Fluid. Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.

17.1.5. Section 9.4: QA/QC - Matrix Spikes. Section 8.2 of the source method states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Further, Section 8.2.3 of the source method also states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The standard TestAmerica QAM is designed to address the performance monitoring of analytical methodology through the LCS program. A minimum of one MS and MSD will be prepared for each TCLP leachate batch. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results have immediate bearing only on the specific sample spiked and not all samples in the batch.

17.1.6. Section 8.2.2 of the source method states that "In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level." The method also states "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 five times the method detection limit". For several analytes, spiking at the regulatory level is inappropriate to the range of analysis afforded by the determinative methods. Due to the wide range in these levels, TestAmerica spikes at the levels specified in the determinative SOPs.

APPENDIX A

TABLES

Table 3 - Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)	
Contaminant	**mg/L**
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

APPENDIX B

FIGURES

Figures 1 and 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

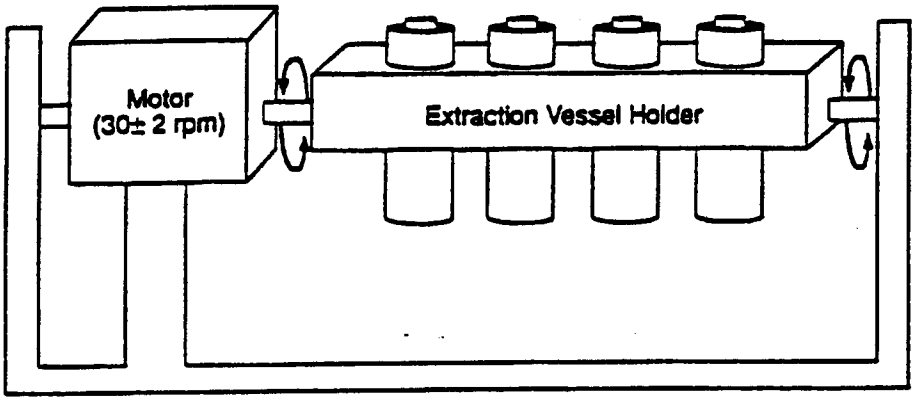

Figure 1. Rotary Agitation Apparatus

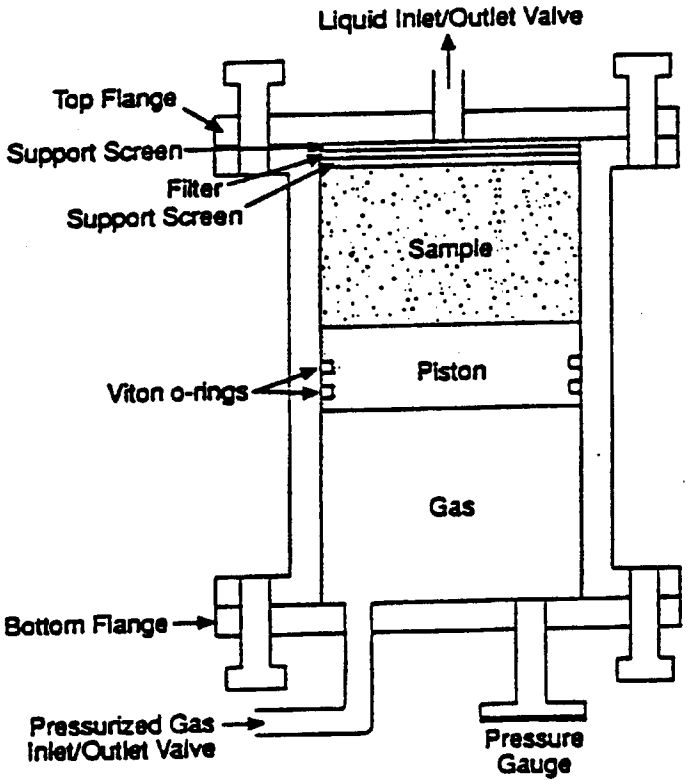

Figure 2 – Cross Section of Zero Headspace Extraction Vessel (ZHE)

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 1

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992

SUBJECT: Notes on RCRA Methods and QA Activities

From: Gail Hansen, Chief
Methods Section (OS-331)

This memo addresses the following topics:

- o 1992 Symposium on Waste Testing and Quality Assurance
- o SW-846 Update
  - Final Rule for January 23, 1989 Proposed Rule
  - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- o Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- o Environmental Monitoring Methods Index (EMMI)
- o Sampling Work Group Formation
- o MICE Update
- o Oily Waste Analysis
- o Electronic SW-846 Availability.

Printed on Recycled Paper

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Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10 (cont'd)

Oily Waste Analysis

One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

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APPENDIX C

LOGBOOK SHEETS

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TESTAMERICA NORTH CANTON ZHE LOGBOOK

Pre-Prep Batch	Sample No.	Ext. Vessel ID	Matrix	Sample Wt (g)	Ext. Buffer ID	Date On	Time On	Analyst On	Date Off	Time Off	Initials Off	Tumbler ID	Tumbler Speed 30±2 RPM	Ext. Fluid ID	Initial ZHE Pressure	Final ZHE Pressure	Comments

TESTAMERICA NORTH CANTON ZHE LOGBOOK - MULTIPHASE

	A	B	C	D	E	F		G	H	
Sample Number	Weight of Container + Sample (g)	Weight of Container + Residue (g)	Difference (A – B) Sample Weight (g)	Weight of the Syringe (or vials) (g)	Weight of the Syringe (or vials) + Filtrate (g)	Difference (E – D) Filtrate Weight (g)	Volume of Filtrate* (mL)	Difference (C – F) Wet Solids Weight (g)	G x 20 Buffer Amount (mL)	Initials

* Filtrate volume only needed when filtrate is incompatible with leachate

TESTAMERICA NORTH CANTON TCLP LOGBOOK

				Pre-Test			% Dry Solids				Leach Start						Leach Finish					
Pre-Prep Batch	Sample No.	Parameters	Matrix	Wt (g) Sample	pH after water	pH after heating	Wt (g) Sample	Wt (g) Filter	Filter + Particulates Wt (g)	% Dry Solids	Wt (g) Sample	Buffer # / ID	Date On	Time On	pH On	Analyst On	Date Off	Time Off	pH Off	Initials Off	Tumbler ID	Comments

TESTAMERICA NORTH CANTON TCLP LOGBOOK – MULTIPHASE

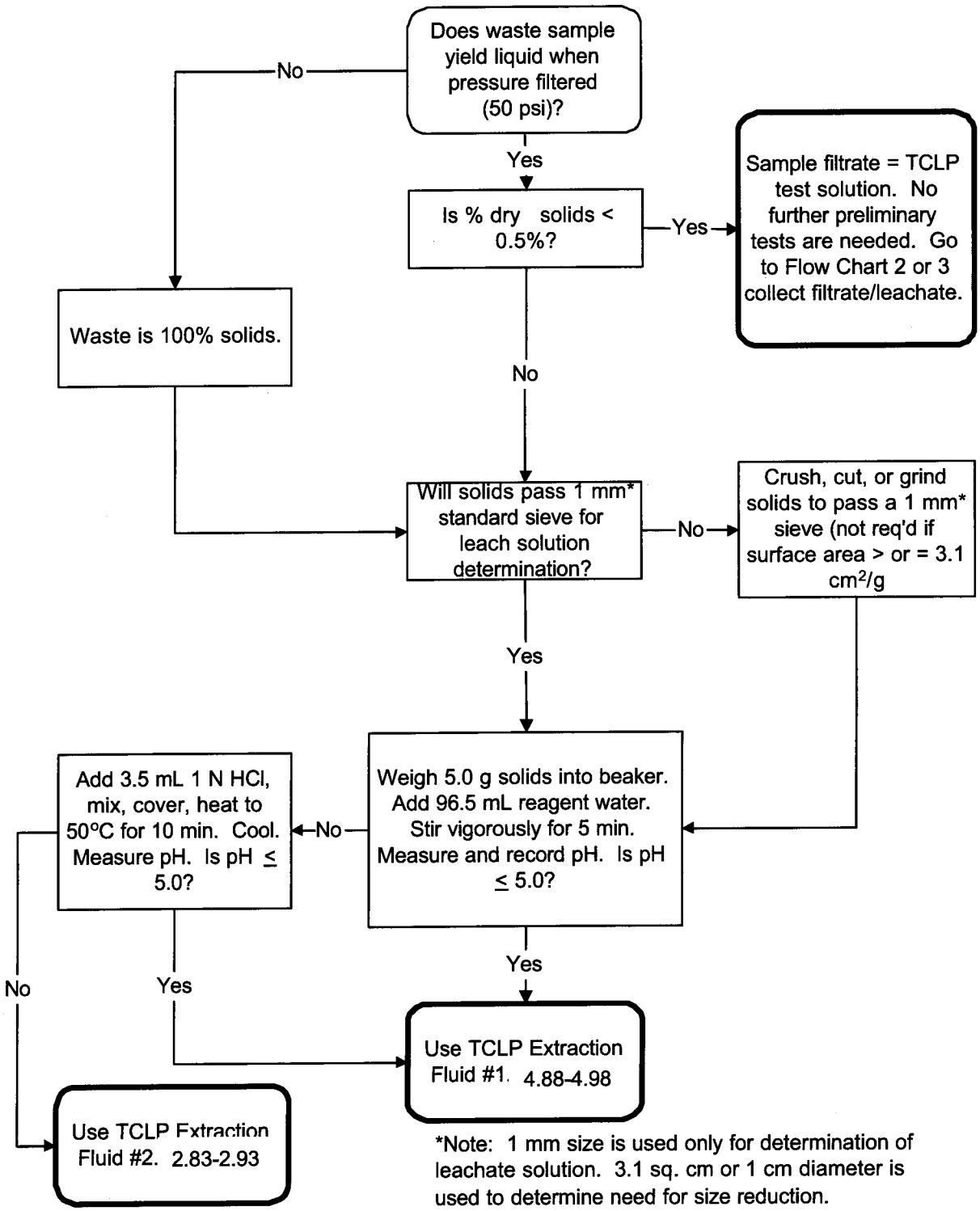
	A	B	C	D	E	F		G	H		
Sample Number	Weight of Container + Sample (g)	Weight of Container + Residue (g)	Difference (A – B) Sample Weight (g)	Weight of the Filtrate Bottle (g)	Weight of the Filtrate bottle + Filtrate (g)	Difference (E-D) Filtrate Weight (g)	Volume of Filtrate* (mL)	Difference (C-F) Wet Solids Weight (g)	G x 20 Buffer Amount (mL)	Initials	Comments

* Filtrate volume only needed when filtrate is incompatible with leachate

APPENDIX D

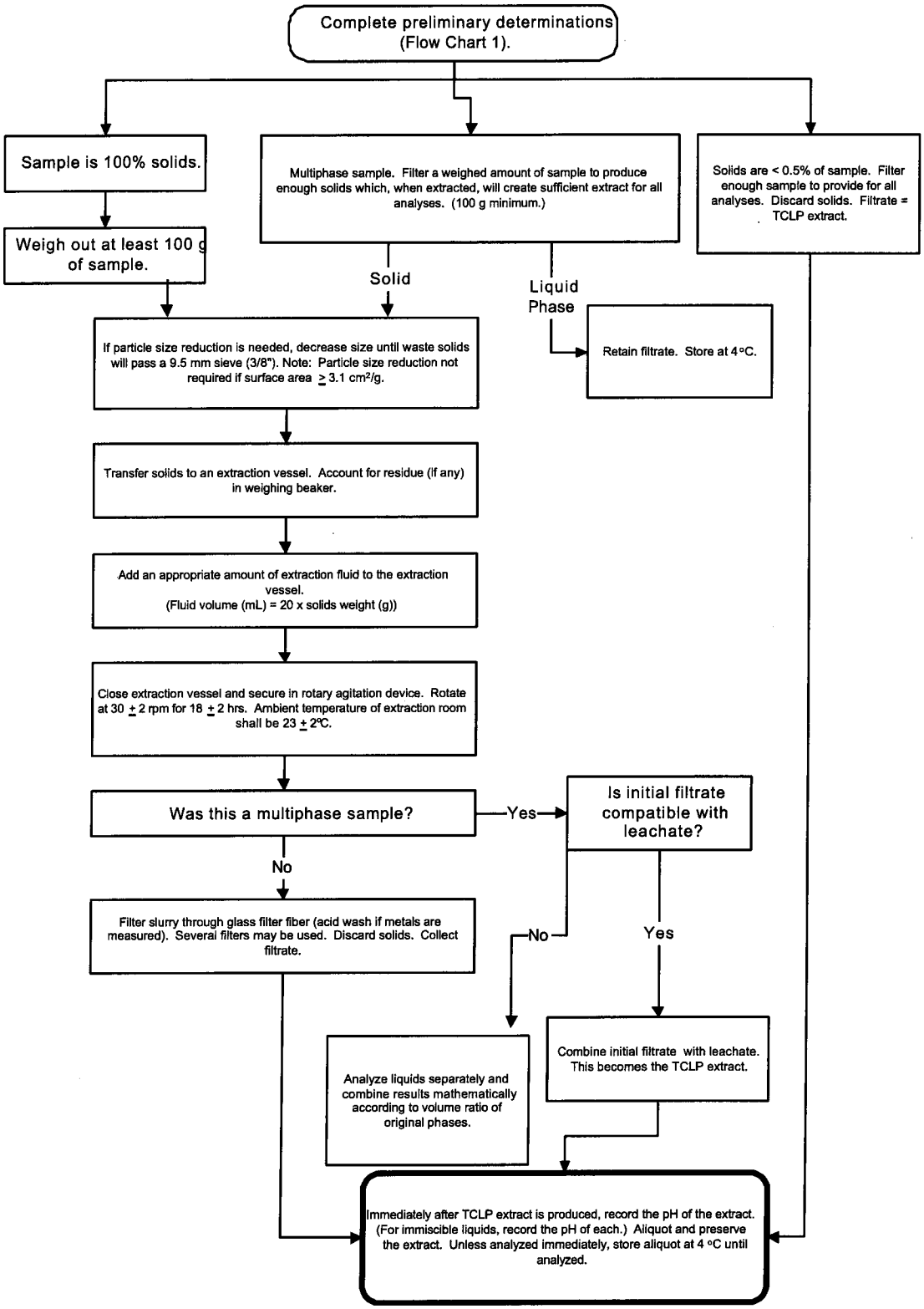
FLOW CHARTS

Flow Chart 1. Preliminary Sample Evaluation (Section 11.2)

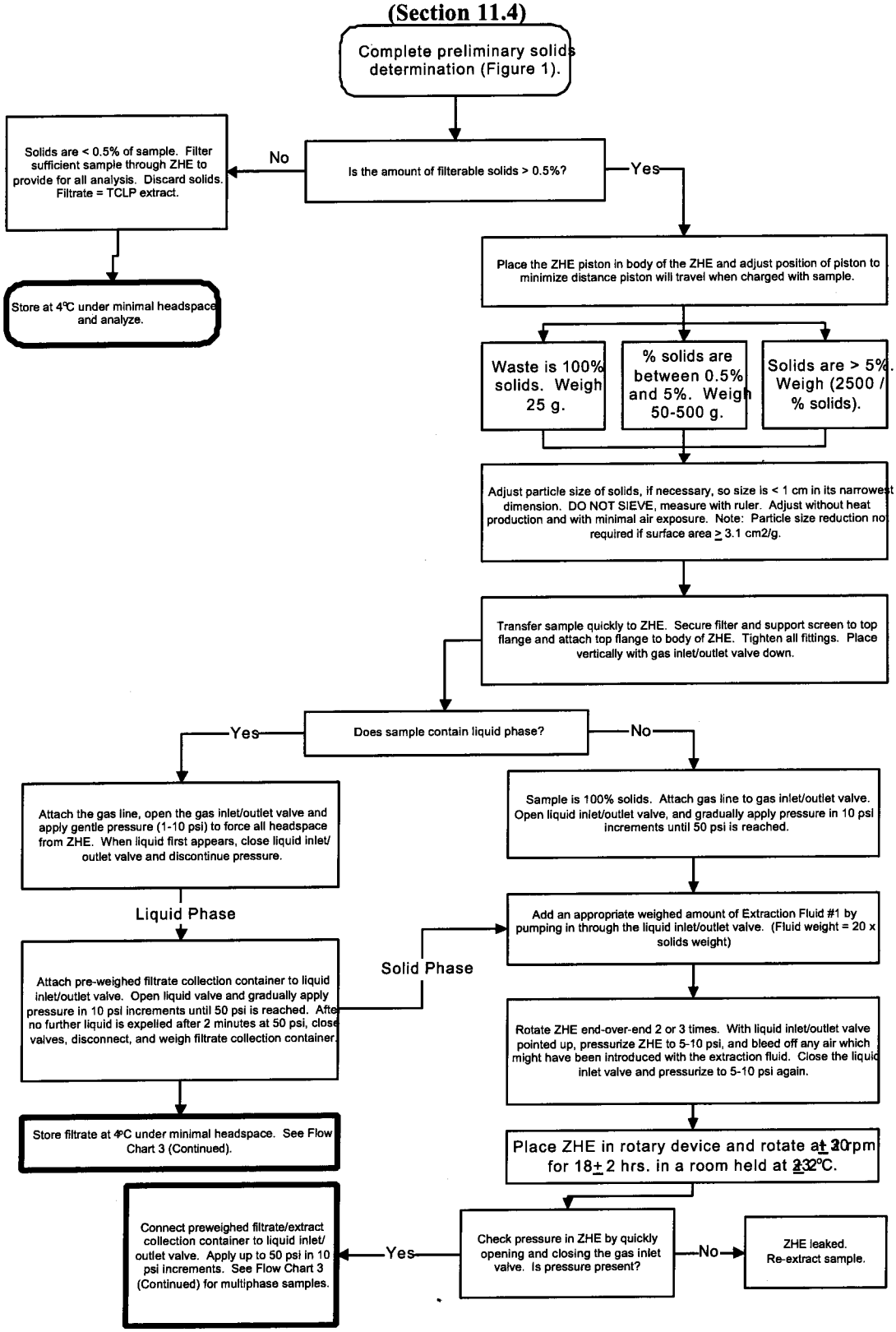


*Note: 1 mm size is used only for determination of leachate solution. 3.1 sq. cm or 1 cm diameter is used to determine need for size reduction.

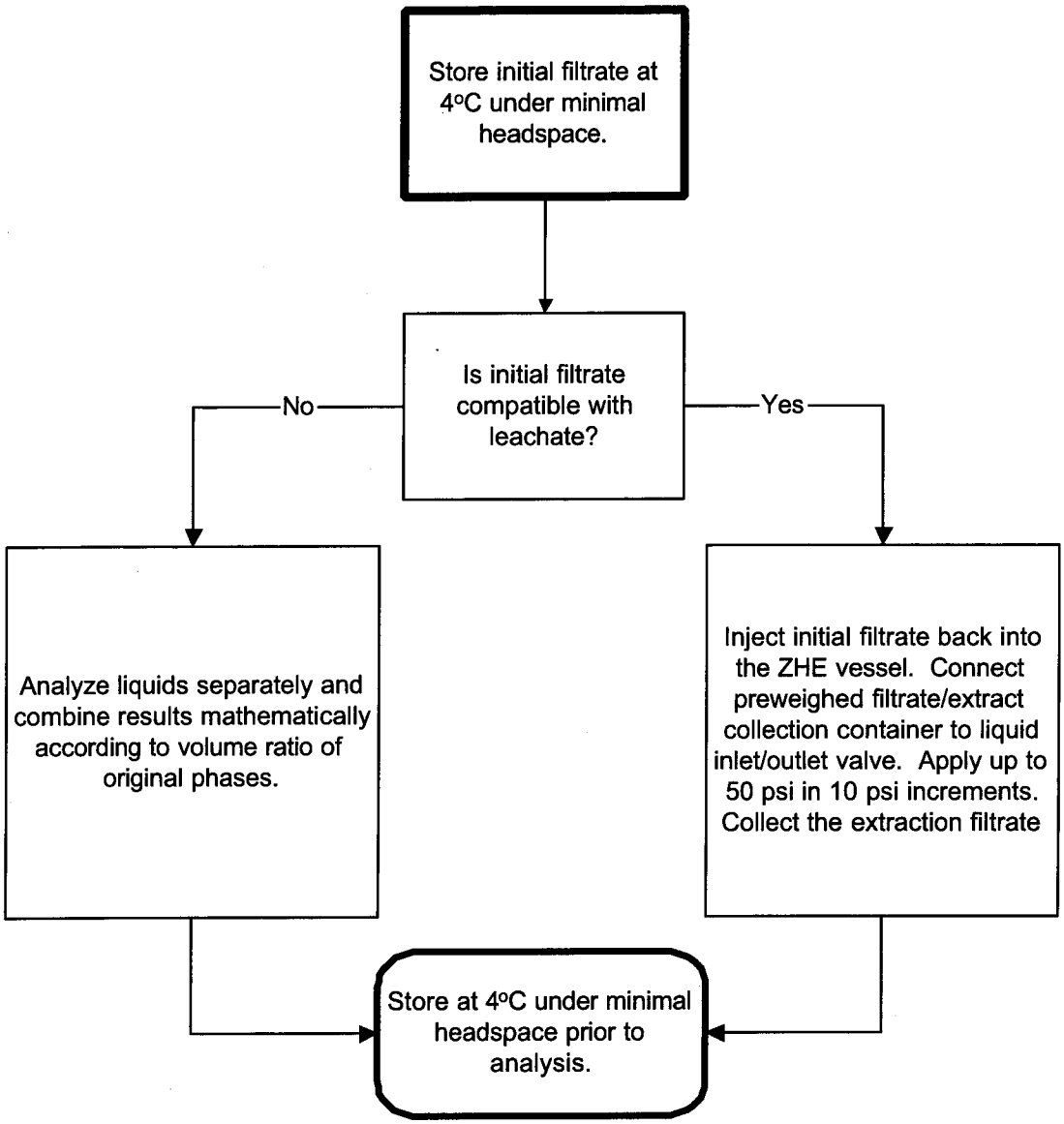
Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



Flow Chart 3. ZHE Extraction, Volatile Constituents (Section 11.4)



Flow Chart 3. ZHE Extraction (Continued)



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TestAmerica North Canton

SOP No. CORP-IP-0003NC, Rev. 1.6
Effective Date: 02/07/07
Cover Page

Title: Acid Digestion for Aqueous Samples
[Method: SW846 and MCAWW 200 Series Methods]

Approvals (Signature/Date):

Technology Specialist	12-14-07 Date	Health & Safety Coordinator	12-14-07 Date
Quality Assurance Manager	12/13/07 Date	Laboratory Director	12/14/07 Date
Technical Director	12/14/07 Date		

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STL STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY SW846 AND MCAWW 200 SERIES METHODS

(Supersedes: Revision 1.5, Dated 12/07/04)

Reviewed by:	Roger K. Toth Technology Specialist	3-6-07 Date
Reviewed by:	Dorothy J. Leeson Quality Assurance Manager	3/7/07 Date
Reviewed by:	William J. Beebe Environmental Health and Safety	2-27-07 Date
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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICP/MS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, 3010A, and 3020A.

1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II (Appendix A). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, certain aqueous sludges, and leachates/extracts.

1.4. SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP.

1.5. MCAWW Method 200.7 is used to prepare surface water, domestic and industrial waste samples for total recoverable and dissolved metals determination by ICP.

1.6. SW-846 Method 3010A is used to prepare aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.

1.7. MCAWW Method 200.7 is used to prepare surface water and wastes that contain suspended solids for total metals analysis by ICP.

1.8. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.9. SW846 Method 3020A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP/MS.

1.10. MCAWW Method 200.8 is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP/MS.

2. SUMMARY OF METHOD

2.1. Method 3005A / Method 200.7 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP Spectroscopy

2.1.1. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A / Method 200.7 - Preparation for Total Metals Analysis by ICP Spectroscopy

2.2.1. A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary) and brought up to volume.

2.3. Method 3020A/Method 200.8 – Preparation for total recoverable or dissolved metals analysis by ICP/MS.

2.3.1. A representative aliquot of sample is heated with nitric acid and until the digestate has been reduced to a low volume. The sample is cooled, filtered (if necessary), and brought up to volume.

3. DEFINITIONS

Additional definitions of terms used in this SOP may be found in the glossary of the LQM.

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix B for additional contamination control guidelines.

4.3. Boron and silica from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. 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.
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.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.8. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9. Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.10. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block or other adjustable heating source capable of maintaining a temperature of 90-95°C.

6.2. Calibrated thermometer that covers a temperature range of 0-200°C.

6.3. Griffin beakers of assorted sizes or equivalent.

6.4. Watch glasses, ribbed or equivalent.

6.5. Whatman No. 41 filter paper or equivalent.

6.6. Funnels or equivalent filtration apparatus.

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repipetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0 - 6).

6.14. Plastic digestate storage bottles.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP LCS/MS spike solution: Prepare the ICP LCS/MS working spike solution from custom stock standards to the final concentration listed in Table III. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months.

7.4. The ICP/MS LCS/MS spike solution is provided directly by the vendor. No further standard preparation is necessary.

7.5. The TCLP MS working spike solution is provided directly by the vendor, no further standard preparation is necessary. Refer to Table V for final digestate spike concentrations.

7.6. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.7. Aqueous laboratory control samples (LCSW) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables III and IV (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and ICP/MS LCS and matrix spike preparations.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.9. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.11. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. For dissolved metals analysis, the samples should be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field. In the event that samples are not field filtered, filtration occurs in the laboratory prior to preparation.

Note: If a sample being analyzed for dissolved metals is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

9. QUALITY CONTROL

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using any method contained within this SOP the following requirements must be met:

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in STL QA Policy S-Q-003 and SOP NC-QA-0021. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOP (CORP-MT-0001).

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs. In cases where there is insufficient sample volume to perform an MS/MSD, an LCS/LCS duplicate is required.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count for determining the size of a preparation batch. MS/MSD are not included in the sample count unless there are multiple sets of MS/MSD per batch. In other words, the first MS/MSD are not counted; all additional MS and MSDs are counted as samples.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs; the blank and all associated samples in the batch must be redigested.

9.4.1. Aqueous method blanks are prepared by taking 50 mL of reagent water through the appropriate procedure as described in Section 11.

9.4.2. TCLP method blanks are prepared by taking 50 mL of leachate fluid through the appropriate procedure as described in Section 11.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Refer to Section 7.3 and 7.4 for instructions on preparation of the aqueous LCS spike solution.

9.5.1. The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water with 1.0 mL for ICP and 0.5 mL for ICP/MS of the working LCS/MS spike solution (Sections 7.3 or 7.4). The LCS is then processed through the appropriate procedure as described in Section 11.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred.

9.6.1. The aqueous matrix spike sample is prepared by spiking a 50 mL aliquot of a sample with 1.0 mL for ICP and 0.5 mL for ICP/MS of the working LCS/MS spike solution (Sections 7.3 or 7.4). The matrix spike sample is then processed as described in Section 11.

9.6.2. The TCLP matrix spike sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution (Section 7.5). The matrix spike sample is then processed as described in Section 11.

NOTE: The TCLP matrix spike must be added prior to preservation of the leachate.

9.6.3. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria.

10. CALIBRATION AND STANDARDIZATION

10.1 The hotplate/hotblock temperature must be verified daily for each hotplate used, and must be recorded on a hotplate/hotblock temperature log.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. All digestion procedures must be carried out in a properly functioning hood.

11.4. All samples are to be checked out of Sample Control with an electronic chain of custody.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.

11.6. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.7. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.8. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.9. The following procedure must be followed for all aqueous sample preparations.

11.9.1. Mix sample by shaking the container.

11.9.2. Measure and transfer 50 mL of the sample into a beaker.

11.9.3. Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with the appropriate spiking solutions (Sections 7.3-7.5 and 9.6).

11.9.4. Measure and transfer 50 mL of reagent water into a beaker for the method blank.

11.9.5. Measure and transfer 50 mL of reagent water into a beaker for the LCS and add the appropriate spiking solutions (Sections 7.3-7.5 and 9.6).

11.10. **Method 3005A / Method 200.7 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP**

11.10.1. To the sample container, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

11.10.2. Cover with ribbed watch glass.

11.10.3. Heat at 90-95°C until volume is reduced to between 15 and 20 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.10.4. Cool the beaker in a fume hood.

11.10.5. Filter sample, if insoluble materials are present, through Whatman 41 filter paper.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.10.6. Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.10.7. Adjust the final volume/mass to 50 mL or 50 g with reagent water. The sample is now ready for analysis

11.11. **Method 3010A / Method 200.7 - Preparation for Total Metals Analysis by ICP Spectroscopy**

11.11.1. To the sample container, add 3.0mL of concentrated HNO_3.

11.11.2. Cover with ribbed watch glass.

11.11.3. Place container on hotblock 90-95°C, and evaporate for 4-5 hours or to low volume of 15-20 mL, while ensuring that no portion of the bottom of the beaker is allowed to go dry.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.11.4. Add 5 mL of 1:1 HCl.

11.11.5. Cover and reflux for an additional 15 minutes to dissolve precipitate or residue. Cool in a fume hood.

11.11.6. Filter sample, if insoluble materials are present, through Whatman 41 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.11.7. Rinse container and filter paper with reagent water to ensure complete sample transfer.

11.11.8. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

11.12. **Method 3020A / Method 200.8 - Preparation for Total or Total Recoverable Metals by ICP/MS**

11.12.1. To the sample container, add 1.5 mL of concentrated HNO_3.

11.12.2. Cover with ribbed watch glass.

11.12.3. Place beaker on hotplate 90-95°C and evaporate to low volume of 15-20 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.12.4. Filter sample, if insoluble materials are present, through Whatman 41 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.12.5. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.12.6. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

11.12.7. ready for analysis.

12. DATA ANALYSIS AND CALCULATIONS

Not Applicable.

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. In general, the matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in determinative SOPs. The laboratory control samples should recover within 20% of the true value until in house control limits are established. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration

study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

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

15.2.1.1. Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in the designated container labeled "Acid Waste".

15.2.1.2. Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container identified as "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A and 3020A.

16.1.2. Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983.

16.1.3. CORP-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010A, 6010B, and Method 200.7.

16.1.4. Corporate Quality Management Plan (QMP), current version.

16.1.5. STL Laboratory Quality Manual (LQM), current version.

16.1.6. STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA-003, STL QC Program.

16.2.2. QA-004, Rounding and Significant Figures.

16.2.3. Glassware Washing, NC-QA-0014

16.2.4. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.5. Method Detection Limits and Instrument Detection Limits, S-Q-003 and NC-QA-0021

16.2.6. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.7. Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference methods.

17.1.1. Modifications applicable to SW-846 reference methods.

17.1.1.1. Chapter 1of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank.

17.1.1.2. The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "flexibility to alter digestion volumes is addressed and "allowed" by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..." EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated " As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

17.1.2. Modifications Specific to Method 3010A

17.1.2.1. Section 11.12.3 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.2.2. The scope of 3010A has been expanded to include silver based on comparison studies with 7760A. Method 3010A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water and TCLP leachate) up to a concentration of 1 ppm silver.

17.1.3. Modifications Specific to Method 3020A

17.1.3.1. Section 11.13.3 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.4. Modifications Specific to MCAWW Methods

It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 11.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness vs. an exact volume).

17.2. Documentation and Record Management

17.2.1. The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst name, matrix, prep type (ICP or ICP/MS), Method reference.
- Sample ID with initial weight/volume and final weight/volume.
- Standards documentation (source, lot, volume added).
- Analyst
- Reagents

APPENDIX A

TABLES

TABLE I. Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A	3020A
Aluminum	Al	7429-90-5	X	X	
Antimony	Sb	7440-36-0	X		
Arsenic	As	7440-38-2	X	X	
Barium	Ba	7440-39-3	X	X	
Beryllium	Be	7440-41-7	X	X	X
Cadmium	Cd	7440-43-9	X	X	X
Calcium	Ca	7440-70-2	X	X	
Chromium	Cr	7440-47-3	X	X	X
Cobalt	Co	7440-48-4	X	X	X
Copper	Cu	7440-50-8	X	X	
Iron	Fe	7439-89-6	X	X	
Lead	Pb	7439-92-1	X	X	X
Magnesium	Mg	7439-95-4	X	X	
Manganese	Mn	7439-96-5	X	X	
Molybdenum	Mo	7439-98-7	X	X	X
Nickel	Ni	7440-02-0	X	X	
Potassium	K	7440-09-7	X	X	
Selenium	Se	7782-49-2	X	X	
Silver	Ag	7440-22-4	X	X	
Sodium	Na	7440-23-5	X	X	
Thallium	Tl	7440-28-0	X	X	X
Vanadium	V	7440-62-2	X	X	X
Zinc	Zn	7440-66-6	X	X	

X - Designates that the preparation method is approved for an element.

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE II. Approved Preparation Method Analytes – NPDES

ELEMENT	Symbol	CAS Number	200.7 (9.4)	200.7 (9.3)
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	X
Arsenic	As	7440-38-2	X	X
Boron	B	7440-42-8	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silicon	Si	7631-86-9	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE III. ICP Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/ MS Level * (ug/l)
Aluminum	100	2000
Antimony	25	500
Arsenic	100	2000
Barium	100	2000
Beryllium	2.5	50
Cadmium	2.5	50
Calcium	2500	50000
Chromium	10	200
Cobalt	25	500
Copper	12.5	250
Iron	50	1000
Lead	50	500
Magnesium	2500	50000
Manganese	25	500
Molybdenum	50	1000
Nickel	25	500
Potassium	2500	50000
Selenium	100	2000
Silver	2.5	50
Sodium	2500	50000
Thallium	100	2000
Vanadium	25	500
Zinc	25	500
Boron	50	1000
Tin	100	2000
Titanium	50	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.3) to 50 mL of sample.

TABLE IV. ICP/MS Aqueous LCS and Matrix Spike Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)
Aluminum	100	1000
Antimony	10	100
Arsenic	10	100
Barium	10	100
Beryllium	10	100
Cadmium	10	100
Calcium	100	1000
Chromium	10	100
Cobalt	10	100
Copper	10	100
Iron	100	1000
Lead	10	100
Magnesium	100	1000
Manganese	10	100
Molybdenum	10	100
Nickel	10	100
Potassium	100	1000
Selenium	10	100
Silver	10	100
Sodium	100	1000
Strontium	10	100
Thallium	10	100
Vanadium	10	100
Zinc	10	100
Boron	10	100
Tin	10	100
Titanium	10	100
Zirconium	10	100

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (7.4) to 50 mL of sample.

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)*
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (7.4) to 50 mL of sample.

TABLE VI. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - CORP-MT-0001	Redigest and reanalyze samples associated with the method blank.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - CORP-MT-0001	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: - NC-MT-0002 - CORP-MT-0001	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: - NC-MT-0002 - CORP-MT-0001	See Corrective Action for Matrix Spike.

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Title: INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES

[SW846 Method 6010B and EPA Method 200.7]

Approvals (Signature/Date):

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Technical Director	Date		

This SOP was previously identified as SOP CORP-MT-0001NC, Rev 3.4, dated 01/08/04

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B and EPA Method 200.7. Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.3. Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, biological, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The

position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.2. Refer to CORP-IP-0002NC, Acid Digestion of Soils, SW846 Method 3050B and CORP-IP-0003NC, Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they

contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. 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.			

5.3.1. The plasma emits strong UV light and is harmful to vision. **NOTE: AVOID looking directly at the plasma.**

5.3.2. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator

6.3. Argon gas supply, welding grade or equivalent

6.4. Coolflow or appropriate water cooling device

6.5. Peristaltic Pump

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes – ranging from 5 µL → 10 ml

6.7. Class A volumetric flasks – range from 50 ml → 2000 ml

6.8. Autosampler tubes – range from 8 ml → 14 ml

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.3. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.4. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.5. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation.

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

9.1.1. Prior to analysis of any analyte using either Method 200.7 or Method 6010B, the following requirements must be met.

9.1.2. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used for each instrument. The IDL must be determined annually. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a blank solution, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the TestAmerica North Canton reporting limit.

9.1.3. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. Refer to TestAmerica North Canton SOP NC-QA-0021 and S-Q-003 for details on MDL analysis and criteria.

9.1.4. Linear Range Verification (LR) - The linear range must be verified every six months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to verify the linear range limit must be analyzed during a routine analytical run and must read within 10% of the expected value.

For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range.

One standard should be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

9.1.5. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.6. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., ICP-MS). Published wavelength tables (e.g., MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I, IA or II. For elements with a reporting limit of 10 ug/L or less, the signal should be ± 2X the RL. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the actual concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response. Additional spectral interference is present from easily ionizable elements such as potassium and sodium in axial viewing instruments.

9.1.7. Rinse Time Determination - Rinse times must be determined upon initial set-up of an ICP instrument. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.1.4) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level). For Ohio VAP projects, all analytes must be less than the reporting limit.

- If the analyte is a common laboratory contaminant (copper, iron, lead, or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. Such action must be addressed in the project narrative.

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be addressed in the project narrative.

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative.

- For dissolved metal samples, which have not been digested, a CCB result is

reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Unless in-house control limits are established, a control limit of 80 - 120% recovery must be applied.
- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The LCSD recovery is evaluated using the same control limits as the LCS. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.
- In the instance where the LCS recovery is greater than the upper control limit and the sample results are < RL, the data may be reported with qualifiers. Such action must be addressed in the report narrative.
- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.
- For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of

that analyte must be in control for the LCS. For both methods 200.7 and 6010B, control limits of 75-125% recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as NC, MSB (i.e., not calculated). Two other narrative notes for metals analyses: Matrix spike/spike duplicate spike recovery/recoveries was/were outside the acceptance limits of some analytes. The acceptable LCS analysis data indicated that the analytical system was operating within control and this condition is most likely due to matrix interference. See the Matrix Spike Report for the affected analytes which will be flagged with N. Matrix spike/spike duplicate relative percent difference (RPD) exceeded the acceptance limits for some analytes. The imprecision may be attributed to sample heterogeneity. See the Matrix Spike Report for the affected analytes, which will be flagged with *.

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists and the data is flagged.

9.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.8 or 11.11

for required run sequence).

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the sample run. The CCV is be a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See Section 11.8 or 11.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, all affected samples will be re-analyzed with valid CCV/CCB pairs. (Refer to Section 11.13 for an illustration of the appropriate rerun sequence). Exceptions: If CCB > RL, samples < RL can be reported with an NCM. If CCV is outside of criteria on the high side, samples < RL can be reported with an NCM.

9.8. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.8.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.8.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 11.10 or 11.13 for required run sequence.)

9.8.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.8.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the TestAmerica North Canton guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs> 10 µg/L must fall within the TestAmerica North Canton guidelines of ± 1x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of

contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

- If the affected element was not required then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.9. CRI - To verify linearity near the RL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run. Additionally, some projects may require CRI analysis at the end of the run. (See Section 11.10 or 11.13 for required run sequence.) Evaluate associated samples based upon advisory limits of +/- 50% of true value.

Note: The custom CRI mix contains most analytes at a level near the standard lab reporting limit.

9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the instructions in Appendix G.

10.2. Profile and calibrate the instrument according to the instrument manufacturer's

recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to Appendix G for detailed set up and operation protocols. Refer to Instruction Manuals in laboratory.

10.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

11.1. For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

11.2. A minimum of <u>two</u> <u>exposures</u> for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.5 it can be demonstrated that a shorter rinse time may be used.

11.4. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.5. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.6. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.

11.7. The use of an internal standard is **<u>required</u>** on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO_3	% HCl
SW846 3050	10	10
SW846 3005	2	5
SW846 3010	6	5

The following procedural guidelines must be followed when using an internal standard:

11.7.1. Typically used internal standards are: yttrium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.7.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards must be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.7.3. The concentration of the internal standard must be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.7.4. The internal standard raw intensity counts must be printed on the raw data.

11.7.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte). The instrument automatically adjusts sample results based on comparison of the internal standard intensity in the sample to the internal standard intensity at calibration.

11.7.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.7.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be diluted and reanalyzed.

11.8. The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CRI (The CRI counts as a sample analysis.)
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Methods 6010B and 200.7 quality control criteria.

11.9. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.10. The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 9.7.

Original Run: Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
10 samples **
CCV *
CCB *

* Failure occurs at CCV/CCB
**Samples requiring rerun for affected analytes

10 samples **
CCV
CCB
10 samples
CCV
CCB

11.11 The instrument may be reprofiled between CCV/CCB pairs to correct for environment induced drift.

11.12 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.13 All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. If an inter-element correction exists for an analyte, which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.14 For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

1) Recovery of the analyte in the matrix spike is not at least 50%,

2) The concentration of the analyte does not exceed the regulatory level, and,

3) The concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix C provides guidance on performing MSA analyses.

11.15 Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager.

11.16 Nonconformance documentation shall be filed in the project file.

11.17 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.18 Analytical Documentation

11.18.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.18.2 All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.18.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.18.4 Sample results and associated QC are entered into the LIMs after final technical review.

12 DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg / L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg / Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:
C = Concentration (mg/L) from instrument readout (mean of two exposures)
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Trivalent Chromium

12.10.1. Trivalent chromium (CR^{+3}) is the result obtained by subtracting the hexavalent chromium (CR^{+6}) results for a sample from the total chromium result from that sample. The total chromium result is determined using the procedures in this SOP. The hexavalent chromium result is determined using the procedures in TestAmerica North Canton SOP NC-WC-0024.

12.10.2. Reporting Limits

12.10.2.1. The TestAmerica North Canton water reporting limit for trivalent chromium is 0.02 mg/l.

12.10.2.2. The TestAmerica North Canton solid reporting limit for trivalent chromium is 2.0 mg/kg, wet weight.

12.10.3. Calculations: $Cr^{+3} = Cr, total - Cr^{+6}$

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery should fall within +/- 25 % and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to 10xIDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value or within the laboratory's historical acceptance limits.

13.4. Training Qualification:

13.4.1 The Group/Team Leader or the Supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.2. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15.2. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.3. Waste Streams Produced by this Method

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

15.3.2 Acid waste consisting of sample and rinse solution - Any sample waste generated must be collected and disposed of in the acid waste drum located in the metals lab.

15.3.3 Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. References

16.1.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

16.1.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.1.3. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

16.1.4. Inductively Coupled Plasma – Atomic Emission Spectrometric Method for Trace Element Analysis of water and wastes Method 200.7, 40 CFR – Chapter I – Part 136 – Appendix C. Electronic version dated September 30, 2002.

16.1.5. Corporate Quality Management Plan (QMP), current version

16.1.6. TestAmerica Laboratory Quality Manual (LQM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. TestAmerica North Canton QC Program, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.6. Hexavalent Chromium (Colorimetric), NC-WC-0024

16.2.7. Acid Digestion of Soils, SW846 Method 3050B, CORP-IP-0002NC

16.2.8. Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods, CORP-IP-0003NC

16.2.10 Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from <u>both</u> Methods 6010B and 200.7.

17.1.1.1. TestAmerca North Canton Laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

17.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a

specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," TestAmerica North Canton has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.3. Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because TestAmerica North Canton laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, TestAmerica North Canton may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At TestAmerica North Canton, matrix interference is determined by evaluating data for the LCS and MS/MSD. TestAmerica North Canton REQUIRES documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7.

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. TestAmerica North Canton labs utilize the IDL definition as defined in Section 9.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method section 9.3.4 specifies that "Analysis of the IPC (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4." TestAmerica North Canton uses a minimum of two exposures.

17.1.2.4. The 40 CFR version of Method 200.7 requires the instrument check standard to agree within ± 5% of expected values. Also, the 40 CFR version requires the interference check sample to be analyzed at the beginning, end, and at periodic intervals throughout the sample run. At TestAmerica North Canton, the instrument check standard equals

the CCV, which must agree within ± 10% of expected values, and the ICSA standards are analyzed only at the beginning of a sample run. TestAmerica's procedures are in line with the Rev. 4.4, May 1994 version of Method 200.7.

17.1.2.5. Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.6. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.7. Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica North Canton has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.7 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B.

17.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two

times the reporting limit in the blank following consultation with the client.

17.1.3.2. Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica North Canton has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.7

for a detailed description of the required corrective action procedures.

APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010B Target Analyte List

Element	Symbol	CAS #	6010B Analyte	200.7 Analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8	X	X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010B Trace ICP Target Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Non-Routine Analyte List

Element	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Tin	2000	2000
Titanium	1000	1000

TABLE IV. Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000
Boron	10000	200	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

* Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for Trace ICP should be at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met		Terminate analysis. Correct the problem. Prepare new standards. Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery RSD dupl. exp < 3% Method 6010B: 90 - 110 % recovery RSD dupl. exp < 5%	Terminate analysis. Correct the problem. Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis. Correct the problem. Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 - 110 % recovery RSD dupl. exp < 5%	Terminate analysis. Correct the problem. Recalibrate and rerun all samples not bracketed by acceptable CCV. Samples < RL can be accepted if the CCV is outside on high side with NCM.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero	Terminate analysis. Correct the problem. Recalibrate and rerun all samples not bracketed by acceptable CCB. Samples < RL can be accepted if the CCB is outside on high side with NCM.
CRI	Beginning of every run	50-150% recovery (advisory)	Evaluate associated samples.
ICSA	Beginning of every run	See Section 9.8.3	See Section 9.8.3
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.8.2.

* See Sections 11.10 and 11.13 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Cont'd)

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
Dilution Test	One per prep batch	For samples > 50x IDL, dilutions must agree within 10%	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL (See 9.2). Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require re-digestion or reanalysis (see Section 9.2).	Re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require re-digestion or reanalysis (see Section 9.3)	Terminate analysis. Correct the problem. Re-digest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples	75 - 125 % recovery or in-house control limits. For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data. No flag required if sample level is > 4x the spike added. For TCLP, see Section 11.17.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20% .	See Corrective Action for Matrix Spike.

APPENDIX B

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7, AND TESTAMERICA NORTH CANTON

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CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND TESTAMERICA NORTH CANTON SOP

EPA 200.7	SW6010B	TestAmerica North Canton SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	N/A	N/A
Laboratory fortified blank (LFB)	N/A	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX C

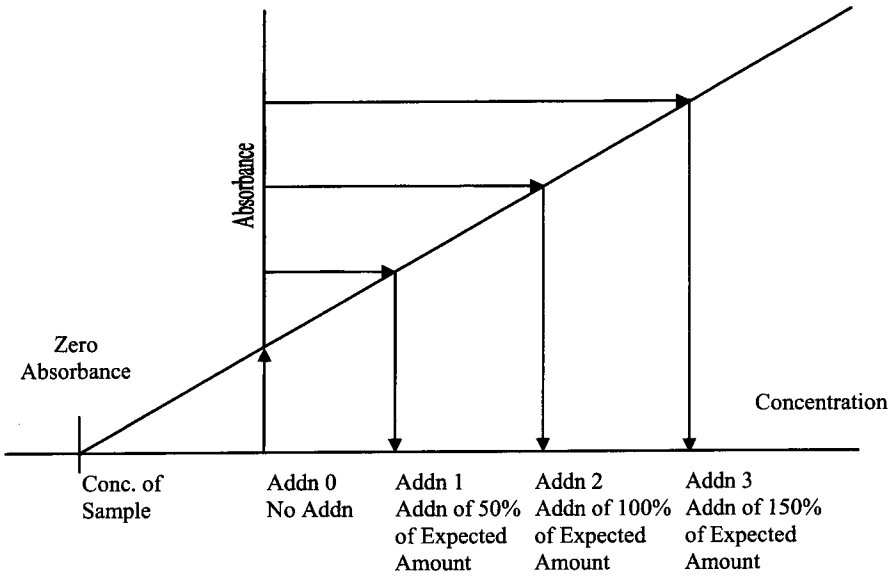
MSA GUIDANCE

APPENDIX C - MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

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APPENDIX D - TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

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APPENDIX E - CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F - PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational:

Daily

Change sample pump tubing and pump windings
Check rinse solution and fill if needed
Check waste containers and empty if needed
Check sample capillary tubing is clean and in good condition
Check droplet size to verify nebulizer is not clogged.
Check sample flow for cross flow nebulizer
Check pressure for vacuum systems

As Needed

Clean plasma torch assembly to remove accumulated deposits
Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance
Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
Apply silicon spray on autosampler tracks
Check water level in cool flow

Bi-yearly

Change oil for vacuum systems
Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

APPENDIX G

ICP OPERATING INSTRUCTIONS

ICP Analysis (TJA 61E)

Example

1. **SETUP**

   a. Plasma Control Panel (enter)

   b. (F1)-Startup

   c. (F9)-Continue

   d. (F2)-Levels

      1. Change auxiliary gas to low – use space bar to toggle

      2. Change nebulizer gas flow to 0.5 L/min.

      3. Change pump rate to 130

      4. Esc

      5. Allow instrument to warm up approximately 30 minutes.

2. **DEVELOPMENT**

   a. Methods (enter)

   b. Enter method name

   c. (F3)-Method Info.

   d. Change file name

   e. (F9)-Done

   f. (F9)-Done/Keep

3. **OPERATION**

   a. Analysis (enter)

   b. (F5)-Profile

      1. (F3)-Automatic

2. (F1)-Run
3. If peak position is greater than +/- .05 units from the center peak position, you must adjust the profile. If it is within +/- .05 units, press (F9)-done.
4. To adjust select (F1)-CalcSS and enter current vernier position. (enter)
5. Adjust to new vernier position(F9)-done
6. Rerun profile until peak position is +/- .05 units.
7. (F9)-Done

c. Autosampler (F9)

1. Enter method name (enter)
2. Enter autosampler table name (enter)
3. (F1)-Run

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Implementation Date 9-10-07

SOP No. NC-MT-0002
Revision No. 4.3
Revision Date: 07/28/07
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TestAmerica North Canton
STANDARD OPERATING PROCEDURE

TITLE: **INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY, EPA METHODS 6020 AND 200.8**

(SUPERSEDES: 4.2 Dated 01/08/04)

Reviewed by:	Roger K. Toth Technology Specialist	8-30-07 Date
Approved by:	Dorothy J. Lusson Quality Assurance Manager	9/5/07 Date
Approved by:	[illegible] Environmental Health and Safety Coordinator	9/4/07 Date
Approved by:	[illegible] Laboratory Director	9/4/07 Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP-MS) based on SW-846 protocol as described in EPA Method 6020 and 200.8. The source method lists fifteen elements approved for analysis by ICP/MS (Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl, and Zn). Additional elements may be included provided that the method performance criteria presented in Section 9 is met. However, project approval may be required from the controlling agencies for compliance testing beyond the fifteen elements included in the promulgated method.

1.2. This procedure also describes the requirements for performing analysis of ground waters, surface waters and drinking water.

1.3. The procedure is applicable to the analysis of waters, soils, and wastes. No digestion is required prior to analysis for dissolved elements in water samples, but the samples must be filtered and preserved prior to analysis. Preliminary acid digestion is required for groundwater, aqueous samples, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are requested. See SOP # CORP-IP-0002NC and SOP #CORP-IP-0003NC for preparation details.

1.4. The associated QuantIMs method codes are MH (6020) and QV (200.8).

1.5. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates, or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into an R.F. plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer capable of providing a resolution better than or equal to 0.9 AMU peak width at 10% of the peak height. For analysis by methods 200.8 the resolution requirement is 1.0 amu at 5% peak height. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier. Interference must be assessed and valid corrections applied, or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents and the constituents of the sample matrix. Recommended elemental equations which correct for many of these interferences are listed in Table I.

Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

3.2. *Dissolved Metals* - Those elements which pass through a 0.45 µm membrane filter (sample is acidified after filtration).

3.3. *Suspended Metals* - Those elements which are retained by a 0.45 µm membrane filter.

3.4. *Total Metals* - The concentration determined on an unfiltered sample following vigorous acid digestion.

3.5. *Total Recoverable Metals* - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.

3.6. *Instrument Detection Limit (IDL)* - See Section 9.1.1.

3.7. *Sensitivity* - The slope of the analytical curve (i.e. the functional relationship between raw instrument signal and the concentration).

3.8. *Tuning Solution* - This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.

3.9. *Initial Calibration Verification/Quality Control Standard (ICV/QCS)* - A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at a concentration other than that used for instrument calibration.

3.10. *Continuing Calibration Verification (CCV).* - A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance.

3.11. *Interference Check Standard (ICS)* - A solution containing both interfering and analyte elements of know concentration that is used to verify background and interelement correction factors.

3.12. *Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB)* - A multi-element

standard of known concentrations which is carried through the entire sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

3.13. *Reagent Blank* - High purity (> 18 megohm-cm) water carried through the entire digestion process.

3.14. *Calibration Blank* - High purity (> 18 megohm- cm) water acidified with the same acid concentrations present in the standards and samples Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

3.15. *Method Detection Limit (MDL)*. See Section 9.1.3

4. INTERFERENCES

4.1. Isobaric Interferences - Isobaric interferences in the ICPMS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software.

4.2. Isobaric Molecular and Doubly Charged Ion Interferences - Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. Table II lists isobaric interferences which might possibly affect required analytes. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied and the data flagged to indicate the presence of interferences.

4.3. Physical Interferences - Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.3.1. Internal standards should be added at a level to give approximately 100,000 – 1,000,000 counts of raw signal intensity. The mass of the internal standard should ideally be within 20 amu of the mass of the measured analyte.

4.3.2. Matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing method 6020 the internal standard intensities must be between 30% and 120% of the intensities in the calibration blank. When performing method 200.8 the internal standards must be between 60% and 125% of the calibration blank If they fall outside this window, a dilution is performed on the sample to correct for matrix effects and the sample reanalyzed.

4.3.3. Memory effects are dependent on the relative concentration differences between samples and/or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

4.3.4. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. 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.

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

5.4. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.8. The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.9. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor and the EH&S Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Argon gas: High purity grade (99.99%).

6.2. Inductively Coupled Plasma Mass Spectrometer capable of providing resolution, less than or equal to 0.9 AMU at 10% peak height from 6-253 AMU and 1.0AMU at 5% peak height from 6-253 AMU with a data system that allows corrections for isobaric interferences and the application of the internal standard technique.

6.3. A four channel peristaltic pump.

6.4. Appropriate water cooling device.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Autosampler with autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Calibration standards are purchased as custom multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. See Table XI.

7.2. Check Calibration Standard (ICV)

A quality control standard similar to the calibration standards and prepared in the same acid matrix. This solution must be made at a concentration near the midpoint of the calibration curve. This standard is composed of analytes from a different source from those used in the calibration of the instrument. See Table XI.

7.3. The tuning solution is purchased as custom multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. The solution must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year.

7.4. The use of hydrochloric and sulfuric acids should be minimized due to higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

7.5. Reagent water:

7.5.1. ASTM Type I or equivalent for the elements of interest, generated using an ion-exchange water polishing system capable of achieving 18.0 megohm-cm.

7.6. Rinse Solution: Carefully dilute 200 mL of concentrated HNO_3 and 40mL of concentrated HCl to 4.0 L with reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous samples are preserved with nitric acid to a pH of 2, and may be stored in plastic or glass. Preservation must be verified prior to analysis.

8.2. Soil samples do not require preservation, but must be stored at 4° ± 2°C until the time of preparation.

8.3. The analytical holding times for metals are six months from the time of collection.

8.4. Solid and aqueous samples must be digested prior to analysis by the appropriate method.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

Table X provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action. Prior to analysis of any analyte the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL). IDLs can be determined by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as a separate analytical sample. The IDL should be performed every three months. The IDL is calculated by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days.

9.1.1.1.IDL = (3) (s), where s = standard deviation.

9.1.2. Linear Calibration Ranges - Linear calibration ranges are primarily detector limited. The linear range must be determined at instrument setup, and the upper limit must be verified annually or whenever a change in instrument hardware or operating conditions, in the judgement of the analyst, may lower expected ranges.

Standards used to determine or verify linear ranges must be analyzed during a routine analytical run. The linear range is the concentration above which sample results cannot be reported.

9.1.2.1. For initial determination of the upper limit of the linear range, determine the signal responses from three different concentration standards across the estimated range. One standard must be at the upper limit of the estimated range. Results must recover within 10% of the expected value for the three standards. The Linear Range is then set at the concentration of the high standard.

9.1.2.2. For verification of the upper limit of the linear range, the high standard must recover within 10% of its expected value

9.1.3. Method Detection Limit (MDL) - The laboratory shall determine a method detection limit for all analytes of interest initially and annually thereafter. The MDL study is performed and calculated according to TestAmerica SOP S-Q-003 and NC-QA-0021, which are based on 40CFR Part 136 Appendix B.

9.2. Batch Definition

9.2.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.3. Method Blank

9.3.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level). **Note:** For Ohio VAP samples, all analytes must be less than the reporting limit.

- If the analyte is a common laboratory contaminant (copper, iron, lead, calcium, magnesium, potassium, sodium or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. Barium, Chromium and Manganese may also be considered common laboratory contaminants at ICPMS reporting limits. **Such action must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative.**

9.3.2. For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB

9.4. Laboratory Control Sample (LCS)

9.4.1. One LCS from an independent source must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The historical limits for the LCS for each analyte are in the LIMS system. If the LCS exceeds these limits for any analyte, that analyte is judged to be out of control and must be corrected before the analysis can be reported

9.4.2. Corrective Action for LCS

9.4.2.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.4.2.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.4.2.3. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable. **Note:** For Ohio VAP samples, the batch must be redigested if the exception in Section 9.4.2.2 is not applicable.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. The historical spike recovery acceptance limits for each analyte are in the LIMS system. If they are not in control and all other quality control criteria have been met, then a matrix interference is suspected.

9.5.2. Corrective action for MS/MSDs

9.5.2.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.5.2.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as NC (not calculated).

9.5.2.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the laboratory limits.

9.5.2.4. If client program requirements specify to confirm matrix interference's, repreparation and reanalysis of the MS/MSD may be necessary.

9.6. Sample Duplicate

9.6.1. A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.6.2. Sample duplicates may be performed in lieu of or in addition to MSDs.

9.7. Control Limits

9.7.1. Control limits are established by the laboratory as described in SOP NC-QA-0018.

9.7.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs (QC Browser program).

9.8. Method Detection Limits (MDLs) and MDL Checks

9.8.1. MDLs and MDL Checks are established by the laboratory as described in SOP NC-QA-0021.

9.8.2. MDLs are easily accessible via the LIMs (QC Browser program).

9.9. ICV/CCV/QCS - Calibration accuracy is verified at the beginning of each analytical run by analyzing a second-source initial calibration verification (ICV) standard. A continuing calibration verification (CCV) standard is analyzed at a 10% frequency throughout the run. The ICV must be within 10% of the expected value, or the analysis is terminated. The CCV must be within 10% of the expected value for method 6020 or 15% of the expected value for method 200.8. Sample results may only be reported when bracketed by valid CCV's.

9.10. RL Verification Standard –An independent standard is analyzed after the ICV to monitor the lab's ability to produce reliable results at RL-level concentrations. There is no set acceptance criteria established for this standard, but generally results should be within 50% of the expected value.

9.11. ICB/CCB/CB - The initial calibration blank must be analyzed immediately following the ICV. The continuing calibration blank must be analyzed at a frequency of 10% throughout the remainder of the analytical run. The ICB/CCB must fall within +/- the

reporting limit from zero.

9.12. Interference Check Solutions (ICSA/ICSAB) ***Method 6020 only***- The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Table V. The interference check solutions must be analyzed at the beginning of every analytical run and every 12 hours thereafter. The results of solution "A" and solution "AB" should be monitored for possible interferences.

9.12.1. Control limits of spiked analytes in the ICSA/ICSAB solution are +/- 50% of true value. Some projects may require control limits of +/- 20% of true value. Control limits of non-spiked analytes are +/- 2x the reporting limit or less than 1 ug/L.

Note: It may not be possible to obtain absolutely clean ICSA/ICSAB standards. If contamination can be confirmed by another method (ICP/GFAA), acceptance criteria will be applied at that level and the data accepted.

9.13. Internal Standards

The intensities of all internal standards must be monitored throughout the run. The internal standard in the samples must be between 30% and 120% of the intensity of the calibration blank for Method 6020, and between 60% and 125% for Method 200.8. If the sample falls outside of this criteria, perform the following procedures. First, evaluate nearby CCVs and CCBs. If sample internal standard recoveries appear to be related to instrument drift, then rerun affected samples. If sample internal standard recoveries appear to be primarily sample or matrix related, then perform appropriate dilutions until the internal standard recoveries are within the method criteria. In no case, may Method 6020 sample results be reported with internal standard recoveries greater than 40% higher than recoveries in surrounding CCVs/CCBs. Alternately, the run may be reprocessed with an alternative internal standard that is not in the samples and at an appropriate mass for the masses being reported.

9.14. Serial Dilution ***Method 6020 only*** - One serial five-fold dilution should be analyzed per batch for each matrix. If the analyte concentration is within linear range of the instrument and sufficiently high (generally, a factor of 100 times above the reporting limit), the serial dilution must agree within 10% of the original analysis. If not, an interference effect must be suspected, the result is flagged and included in the final report narrative. Samples identified as blanks cannot be used for serial dilution.

9.15. Post-Digestion Spike Addition (PDS) ***Method 6020 only*** - If the serial dilution fails to meet the acceptance criteria, a PDS must be performed as follows. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered within 75 -

125% of the known value. If the PDS fails to meet this criterion, matrix interference should be suspected.

9.16. General Corrective Action Requirements - The general requirements for evaluation of QC results and corrective action for failures is described in TestAmerica Policy QA-003

9.17. Nonconformance and Corrective Action

9.17.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument StartUp

Set up the instrument according to manufacturers operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning.

10.2. Instrument Tuning / Mass Calibration / Daily Performance

10.2.1. Daily Performance – Refer to Appendix A for ICPM/MS Instrument Instructions. Instrument manuals are available as needed. Verify instrument performance daily with a solution containing elements representing all of the mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of 4 times. For method 200.8, the tuning solution must be analyzed 5 times with a relative standard deviation less than 5%.

10.2.2. Check mass calibration and resolution daily.

10.2.2.1. Mass Calibration Check – The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

10.2.2.2. Mass Resolution Check - The resolution must be verified to be less than 0.9 amu full width at 10% peak height. Due to a limitation of the instrument software, the resolution requirement for method 200.8 of 1.0 amu full width at 5% peak height cannot be verified automatically. If the mass resolution requirement of 0.9AMU at 10% peak height is met the 200.8 requirement is also satisfied.

10.3. Calibrate the instrument for the analytes of interest according to manufacturer's instructions. Routine calibration and calibration verification levels are shown in Table

XI. The calibration should include a blank and three standards. For a linear multi-point calibration curve, the correlation coefficient must be >/= 0.995. Report the average of at least two integrations for both calibration and sample analysis. A calibration must be performed daily and each time the instrument is set up. Instrument run may be continued over periods exceeding 24 hours as long as calibration verification, interference check, and internal standard QC criteria are met.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Preliminary acid digestion is required for groundwater, aqueous samples, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are requested. See SOP CORP-IP-0002NC and SOP CORP-IP-0003NC for preparation details.

11.4. Sample Analysis

11.4.1. Flush the system with the rinse blank for at least 30 seconds between samples and standards during the analytical run.

11.4.2. Masses which would affect the data quality must be monitored during the analytical run to determine the potential effects of matrix on a given element.

11.4.3. Dilute and reanalyze samples that are more concentrated than the linear range for an analyte or specific isotope of interest. No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the IDL (the sample should be diluted to the approximate midrange of the analytical curve), unless the dilution is for internal standard recoveries.

11.4.4. The analytical run sequence should be performed as follows to meet all quality control criteria:

Warm-up

Verify instrument performance
Calibration blank
Calibration standards
ICV
ICB
RL verification standard
ICSA (6020 Only)
ICSAB (6020 Only)
CCV
CCB
10 Samples
CCV
CCB

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

Note: The mean of 2 exposures is used to derive the sample concentrations used in the calculations in this section.

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = \mathbf{100 \times \left(\frac{Found\ (ICV)}{True\ (ICV)} \right)}$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = \mathbf{100 \times \left(\frac{Found\ (CCV)}{True\ (CCV)} \right)}$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \mathbf{100 \times \left(\frac{SSR - SR}{SA} \right)}$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Note: When sample concentration is less than the method detection limit, use SR = 0 for purposes of calculating % Recovery.

12.4. The relative percent difference (RPD) of sample duplicates are calculated according to the following equation:

$$RPD = \mathbf{100 \times \left[\frac{(DU1 - DU2)}{(DU1 + DU2)/2} \right]}$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$\text{Result (ug/L)} = \mathbf{\frac{(C \times V1 \times D)}{V2}}$$

Where:
C = Concentration from instrument readout, ppb
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The concentration determined in digested solid samples when reported on a wet weight

basis is as follows:

$$\text{Result (ug/kg)} = \frac{(C \times V \times D)}{W}$$

Where:

C = Concentration from instrument readout, ppb
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight, in g, of wet sample digested

12.7. Sample results should be reported according to the following significant figure rules:

12.7.1. All uncorrected values less than the detection limit are reported as "less than" the detection limit.

12.7.2. Positive results for target analytes are reported to three significant figures if the method is used without dilution.

12.8. Positive results obtained after dilution and results for non-certified analytes are reported to two significant figures

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Refer to Table I for the list of analytes that may be analyzed using this SOP for methods 6020 and 200.8. Additional analytes may be analyzed if all method required QC is acceptable.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within historical laboratory control limits and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.3. The laboratory control samples should recover within 20% of the true value until in house control limits are established.

13.4. Training Qualifications:

13.4.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the

required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.4. Waste Streams Produced by the Method

15.4.1. Acid waste consisting of sample and rinse solution generated by this method.

15.4.1.1. Aqueous waste can be poured down the drain if the pH is between 4 and 10. Any sample waste generated that is not in this pH range must be collected and disposed of in the acid waste drum located in the metals lab.

16. REFERENCES

16.1. References

16.1.1. Test Methods For Evaluating Solid Waste, EPA SW-846, 3rd Edition, Final Update II, Method 6020: "Inductively Coupled Argon Plasma - Mass Spectrometry", Revision 0, September 1994.

16.1.2. Environmental Monitoring Systems Laboratory, EPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry", Revision 5.4, EMMC Version

16.1.3. Corporate Quality Management Plan (QMP), current version.

16.1.4. TestAmerica Laboratory Quality Manual (LQM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. CORP-IP-0002NC, Acid Digestion of Soils, SW846 Method 3050B, latest version.

16.2.3. CORP-IP-0003NC, Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods

16.2.4. Glassware Washing, NC-QA-0014

16.2.5. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.6. Method Detection Limits and Instrument Detection Limits, NC-QA-0021 and S-Q-003.

16.2.7. Supplemental Practices for DoD Project Work, Navy/Army SOP, NC-QA-0016.

16.2.8. Standards and Reagents, NC-QA-0017.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. Refer to Table XII for associated reporting limits

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method deviations

17.2.1. Deviations from method 6020

17.2.1.1. Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept in the laboratory.

17.2.1.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit, not 3 times the instrument IDL.

17.2.1.3. Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18megOhm) and by the analysis of blanks.

17.2.1.4. Internal standard recoveries may be less than 80% in CCV's and CCB's as long as QC criteria are met. Sample internal standard recoveries may never be greater than 40% higher than recoveries in associated CCV's/CCB's.

17.2.2. Deviations from method 200.8

17.2.2.1. Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept in the laboratory.

17.2.2.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit, not 3 times the instrument IDL.

17.2.2.3. Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18 megOhm) and by the analysis of blanks.

17.2.2.4. Resolution criteria of the mass calibration is met if the resolution criteria for method 6020 is satisfied.

17.2.2.5.The concentration of most analytes in the LCS is 100 µg/L. This is made from a commercially available stock solution and has all analytes at the same level. Verification records for this solution are kept in the laboratory.

17.2.2.6. Results are reported up to the verified linear range, not up to only 90% of the linear range.

TABLE I: Recommended Elemental Interference Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	(1.0000)(27M*)
Sb	none	(1.0000)(121M)
As	ArCl, Se	(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)
Ba	none	(1.0000)(135M)
Be	none	(1.0000)(9M)
Cd	MoO, Sn	(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)
Ca	none	(1.0000)(44M)
Cr	none	(1.0000)(52M)
Co	none	(1.0000)(59M)
Cu	none	(1.0000)(65M)
Fe	none	(1.0000)(57M)
Pb	none	(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)
Mg	none	(1.0000)(25M)
Mn	none	(1.0000)(55M)
Ni	none	(1.0000)(60M)
K	none	(1.0000)(39M)
Se	Ar2	(1.0000)(78M) - (1.1869)(76M)
Ag	none	(1.0000)(107M)
Na	none	(1.0000)(23M)
Tl	none	(1.0000)(205M)
V	ClO, Cr	(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)
Zn	none	(1.0000)(66M)
6Li	Li (natural)	(1.0000)(6M) - (0.0813)(7M)
Sc	none	(1.0000)(45M)
Y	none	(1.0000)(89M)
Rh	none	(1.0000)(103M)
In	Sn	(1.0000)(115M) - (0.0149)(118M)
Tb	none	(1.0000)(159M)
Ho	none	(1.0000)(165M)
Bi	none	(1.0000)(209M)

* M = Total ion count rate at the specified mass

TABLE II: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	
^{59}Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	SS,SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}

TABLE II: (cont.) Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN				
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

Note: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

Table III: Changes in Isobaric Molecular-Ion Interferences with Changing Plasma Conditions**

	Molecular Interference	Nebulizer		
		High	Average	Low
Oxides:	ScO/Sc	0.00326	0.00055	0.00116
	YO/Y	0.00568	0.00395	0.00353
	TbO/Tb	0.0156	0.00648	0.00614
	ClO, Cl	0.00725	0.00227	0.00233
Hydroxides:	ScOH/Sc	0.00040	0.00011	0.00000
	YOH/Y	0.00078	0.00044	0.00048
	TbOH/Tb	0.00034	0.00008	0.00011
	ClOH/Cl	0.00048	0.00031	0.00029
Chlorine:	ClO/Cl	0.00725	0.00227	0.00233
	ClOH/Cl	0.00048	0.00031	0.00029
	ArCl/Cl	0.00605	0.00091	0.00477

** Information for this table is being determined by the EPA.

Table IV: Recommended Internal Standards

Method 6020	Method 200.8
Li	Sc
Sc	Y
Y	In
Rh	Tb
In	Bi
Tb	
Ho	
Bi	
Ge	

Table V: Interference Check Sample Components and Concentrations

(ICSAB minors are suggested spike levels)

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	50	50
Ca	50	50
Fe	50	50
Mg	50	50
Na	50	50
P	50	50
K	50	50
S	50	50
C	100	100
Cl	500	500
Mo	1.0	1.0
Ti	1.0	1.0
As	0.0	0.1
Cd	0.0	0.1
Cr	0.0	0.1
Co	0.0	0.1
Cu	0.0	0.1
Mn	0.0	0.1
Ni	0.0	0.1
Se	0.0	0.1
Ag	0.0	0.1
V	0.0	0.1
Zn	0.0	0.1

Table VI: Sample Preservation and Holding Times

Measurement Parameter	Container (1)	Preservative (2)	Maximum Holding Time (3)
Waters:			
Metals (4)	P,G	HNO_3 to pH < 2	6 months
Soils/Sediments/Wastes:			
The preservation required for soil/sediment/waste samples is maintenance at 4°C (± 2°C) until digestion.			

Footnotes: (1) Polyethylene (P) or glass (G).

(2) Sample preservation is performed by the sampler immediately upon sample collection.

(3) Samples must be analyzed as soon as possible after collection. The times listed are the maximum times that sample may be held before analysis and still considered valid. Holding times are calculated from the date when the sample was collected.

(4) Samples are filtered immediately on-site by the sampler before adding preservative for dissolved elements.

Table VII: Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. Suggested masses for method 200.8 are in "quotes".

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"75"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"9"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
24, **25**, **26**	Magnesium
"**55**"	Manganese
58, "**60**", 62, **61**, 64	Nickel
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
"**205**", 203	Thallium
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
72	Germanium
139	Lanthanum
118	Tin
35, 37	Chlorine
"98", 96, 92, **97**, 94	Molybdenum

Note: It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

Table VIII: Tuning Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below are two groups of suggested solutions which cover a typical mass calibration range.

Method 6020

Element	Concentration (µg/L)
Solution A	
Mg	10.
Rh	10.
Pb	10.
Solution B	
Li	10.
Co	10.
In	10.
Tl	10.

Method 200.8

Element	Concentration (µg/L)
Be	10.
Mg	10.
Co	10.
In	10.
Pb	10.

Table IX: Suggested Tuning and Response Factor Criteria

Minimum Response from Tuning Solution

With a Peristalic Pump Speed of 12 RPM:

Be	>1,000
Mg	>10,000
Rh	>100,000
Pb	>50,000
Li	>2,000
Co	>20,000
In	>1,000
Tl	>1,000

Suggested Mass Calibration:

Be	9.0122
Mg	23.98
Rh	102.91
Pb	207.98
Li	7.016
Co	58.9332
In	114.904
Tl	204.9744

Table X: Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
ICV/QCS	Beginning of every analytical run.	90 - 110% recovery.	Terminate analysis; correct the problem; recalibrate.
ICB/CB	Immediately after each ICV	The result must be < RL.	Terminate analysis; correct the problem; recalibrate.
CCV	Beginning and end of run and every 10 samples.	**6020-** 90 - 110% recovery **200.8-** 85-115% recovery	If unacceptable, correct the problem recalibrate the instrument, reverify calibration and rerun all samples associated with unacceptable CCV's.
CCB	Immediately following each CCV.	The result must be < RL	If unacceptable, correct the problem recalibrate the instrument, reverify calibration and rerun all samples associated with unacceptable CCB's.
ICSA (6020 Only)	Beginning and every 12 hours.	Monitor for possible interferences.	See Section 9.12
ICSAB (6020 Only)	Immediately following each ICSA.	Monitor for possible interferences.	See Section 9.12

Table X (cont'd): Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
Method Blank/Laboratory Reagent Blank	One per lot of 20 field samples or fewer.	The result must be < RL. Sample results greater than 20x the blank concentration or samples for which the contaminant is < RL, do not require redigestion or reanalysis.	Redigest and reanalyze samples. See Section 9.3 for additional requirements.
Laboratory Control Sample/Laboratory Fortified Blank	One per lot of 20 field samples or fewer.	80-120%, or in-house limits (6020), 85-115% (200.8)	Redigest and reanalyze samples. See Section 9.4.
Serial Dilution (6020 Only)	One per lot of 20 field samples or fewer.	90 – 110% recovery	See section 9.14 for additional requirements.
Post-Digestion Spike (6020 Only)	See section 9.15	75-125% recovery	See section 9.15.
Matrix Spike/Matrix Spike Duplicate	One per lot of 20 field samples or fewer.	Must be within laboratory control limits	See section 9.5 for additional requirements.

Table XI: ICP/MS Calibration and Calibration Verification Checklist

Suggested Levels in µg/L

Element	Calibration			ICV	CCV
	1	2	3		
Aluminum	50	5000	10000	25	50
Antimony	2	50	100	25	50
Arsenic	2	50	100	25	50
Barium	1	50	100	25	50
Beryllium	1	50	100	25	50
Cadmium	0.5	50	100	25	50
Chromium	2	50	100	25	50
Cobalt	1	50	100	25	50
Copper	2	50	100	25	50
Iron	20	5000	10000	25	50
Lead	1	50	100	25	50
Manganese	1	50	100	25	50
Nickel	2	50	100	25	50
Selenium	2	50	100	25	50
Silver	0.5	50	100	25	50
Thallium	1	50	100	25	50
Vanadium	5	50	100	25	50
Zinc	10	50	100	25	50

This procedure has been developed for additional elements. Additional elements may be included in the calibration solution at the appropriate levels. Levels may be adjusted to meet specific regulatory or client programs.

Table XII: Suggested ICP/MS Reporting Limits

Water 6020 and 200.8 Solid 6020 (only)

Compound	RL	Units	RL	Units
Aluminum	50	ug/L	--	--
Antimony	2	ug/L	200	ug/kg
Arsenic	5	ug/L	500	ug/kg
Barium	5	ug/L	500	ug/kg
Beryllium	1	ug/L	100	ug/kg
Cadmium	1	ug/L	100	ug/kg
Chromium	2	ug/L	200	ug/kg
Cobalt	1	ug/L	100	ug/kg
Copper	5	ug/L	500	ug/kg
Iron	20	ug/L	--	--
Lead	1	ug/L	100	ug/kg
Manganese	5	ug/L	500	ug/kg
Molybdenum	2	ug/L	200	ug/kg
Nickel	2	ug/L	100	ug/kg
Selenium	5	ug/L	500	ug/kg
Silver	1	ug/L	100	ug/kg
Thallium	1	ug/L	100	ug/kg
Tin	10	ug/L	1000	ug/kg
Vanadium	5	ug/L	500	ug/kg
Zinc	10	ug/L	1000	ug/kg

APPENDIX A – OPERATION INSTRUCTIONS – P.E. 6100 ICP/MS INSTRUCTIONS

A. Light the plasma and start the peristaltic pump.

1. Allow the instrument to warm up approximately 30 minutes.

B. Daily Performance

1. Open Daily2_asx workplace.
2. Open the sampling tab in the method screen.
3. Select and initialize the autosampler. Click O.K.
4. Click on probe. Click go to rinse. Send probe to tube #8.
5. Allow solution to reach plasma and then click on analyze sample.
6. When analysis is complete, send probe back to rinse.

C. Analysis Setup

1. Open Analysis workspace.
2. Under the method window, open the report tab and type in your report filename. Save the method.
3. Highlight the dataset window. Go to file and select new. Type in the dataset name (normally the same as the filename).
4. Highlight autosampler window. Type in your autosampler locations and samples. Right click under measurement action and select what needs to be analyzed. Right click under method description and select method. Verify that all times and rpms on table are correct. Go to file and click save as. Type autosampler table name (normally the same as the filename).

D. Analyze samples

1. Highlight samples to be analyzed.
2. Left click on analyze batch.

APPENDIX B – OPTIMIZATIONS – P.E. 6100

X-Y Adjustment

Adjusts torch to achieve best intensities. This should be done whenever anything is done to the torch or the cones.

1. Open X-Y_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Allow solution to reach plasma and hit analyze sample.
4. Adjust x and y while watching the signal on the realtime window. Adjust only until signal is at its highest.

NEB Lens

This also is done to get best intensities. It effects the shape and the depth of the plasma. This should be done if your oxides or doubly charged are >3%.

1. Open neb lens power oxides.wrk
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on the Autooptimize tab. Select nebulizer gas flow.
4. Click get analyte list.
5. Make sure solution has reached the plasma and click optimize.
6. When done, save the optimization file.

Autolens

Each element done represents a section of the mass spectrum. Be is on the low end, CO is in the middle and Pb is on the higher end. If there is a problem with any particular section of the spectrum, it may be an indication that this needs done. This should probably be performed on a weekly basis.

1. Open Autolens_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on the autolens tab.
4. Click on clear calibration.
5. Click on get analyte list.
6. Make sure the solution has reached the plasma and click on calibrate.

7. Save the optimization file.
8. In the interactive window, the optimization curve can be printed.

Dual Detector Calibration

This extends the dynamic range of the detector. This should be done on a weekly basis. All analytes of interest must be included in the solution.

1. Open dual detector2.wrk
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Click on dual detector cal tab.
4. Click on clear calibration.
5. Click on get analyte list.
6. Make sure the solution has reached the plasma and click on calibrate.
7. When complete, check to make sure that all r values are at least .999.
8. Save the optimization file.
9. In the interactive window, the plots can be displayed.

Tuning

This adjusts the electronics to assure the accuracy of the mass spec. The resolution adjustment assures that the resolution of each mass of interest is within range. This should be done daily.

1. Open tuning_asx.wrk.
2. Under the method window, go to the sampling tab and send the probe to tube #8.
3. Allow sample to reach the plasma and click on tune mass spec.
4. Make sure measured mass and peak width is within range. If its not, resolution will need adjusted. Adding 30 units will decrease the amu by about 0.1. Subtracting 30 units will increase the amu about 0.1.

General Notes

1. All optimizations can be done with the tuning solution, except the dual detector cal needs the cross cal standard with all the elements of interest.
2. All optimizations can be run at 12 rpm, except the dual detector calibration should be at 24 rpm.

APPENDIX C – ICPMS MAINTENANCE SCHEDULE

Daily

Change sample and internal standard pump tubing and pump windings

Check argon gas supply level

Check rinse solution and fill if needed

Check waste containers and empty if needed

Check sample capillary tubing is clean and in good condition

Check sample flow for cross flow nebulizer

Check pressure for vacuum systems

Check daily performance

As Needed

Clean Sampler and skimmer cones

Clean plasma torch assembly to remove accumulated deposits

Clean nebulizer

Replace sample and internal standard capillary tubing and autosampler sipper probe

Replace drain tubing

Perform necessary optimizations

Clean autolens

Monthly

Inspect air filters; clean or replace as needed

Bi-Yearly

Change oil in vacuum pumps

Check water in coolflow

Controlled Copy
Copy No. _____

Implementation Date: 7-18-07

TESTAMERICA NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1

(SUPERSEDES: REVISION 2.5 DATED 01/08/04)

Approved by:	Technical Specialist	7/17/07 Date
Approved by:	Quality Assurance Manager	7/17/07 Date
Approved by:	Environmental, Health and Safety Coordinator	7-17-07 Date
Approved by:	Laboratory Director	7/18/07 Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1.

1.2. The associated LIMs method codes are BL (Method 245.1) and O8 (Method 7470A).

1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.4. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, TCLP, and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed, however Method 7471A (see CORP-MT-0007NC) is usually the method of choice. All matrices require sample preparation prior to analysis.

1.5. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters and domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.6. The TestAmerica North Canton reporting limit for mercury in aqueous matrices is 0.0002 mg/L except for TCLP or SPLP leachates for which the reporting limit is 0.002 mg/L.

1.7. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of

the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.

3.4. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Potassium permanganate, which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.4. Chlorides can cause a positive interference. Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists

at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.5. Interference from certain volatile organic materials that absorb at this wavelength may also occur. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility.

4.6. Samples containing high concentrations of oxidizing organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low.

4.7. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, and this document.

5.2. 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
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	2 ppm-TWA	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause

	Oxidizer Poison	4 ppm-STEL	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 Compounds	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.
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.
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.			

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened,

transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to a Laboratory Supervisor and the EH&S Coordinator.

5.6. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.7. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders are located outside the laboratory and the gas led to the instrument through approved lines.

5.8. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath capable of maintaining a temperature of 90-95 °C.

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1 Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2 Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3 Peristaltic pump, which can deliver 1 L/min, air.

6.2.4 Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5 Recorder or Printer.

6.2.6 Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the

absorption cell and return. The tubing must be inert and mercury-free.

6.2.7 Drying device to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that achieve the same purpose are also acceptable (i.e., Gortex filter).

6.3. 8oz. HDPE Plastic bottles.

6.4. Nitrogen or argon gas supply, welding grade, or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Thermometer (capable of accurate readings at 95 °C).

6.8. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm) mercury standards (in 10% HNO_3) are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. The calibration standards listed in Table I must be prepared fresh daily from the working standard (7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into 100 mL flasks and diluting to volume with reagent water.

7.5. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7. Nitric acid (HNO_3), concentrated, traces metal grade or better.

7.8. Sulfuric acid (H_2SO_4), concentrated, traces metal grade or better.

7.9. Stannous chloride solution: Add 50 g of stannous chloride and 25 mL of concentrated hydrochloric acid to a 500mL volumetric flask and bring to volume with deionized water.

7.10. Sodium chloride-hydroxylamine hydrochloride solution: Add 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride to every 2000 mL of reagent water.

7.11. Potassium permanganate, 5% solution (w/v): Dissolve 100 g of potassium permanganate for every 2000 mL of reagent water.

7.12. Potassium persulfate, 5% solution (w/v): Dissolve 100 g of potassium persulfate for every 2000 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

9. QUALITY CONTROL

9.1. Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.2. Initial Demonstration of Capability

9.2.1 Prior to the analysis of any analyte using 7470A or the 245.1, the following requirements must be met.

9.2.1.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be predetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the TestAmerica North Canton reporting limit.

9.2.2 Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.2.2.1 Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.3. Preparation Batch - A batch is a group of no greater than 20 samples excuding QC Samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Re-preparation and re-analysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported. **Such action must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits the system is out of control and corrective action must occur.

- In the instance where the LCS recovery is greater than the upper control limit and the sample results are less than RL, the data may be reported. Such action must be addressed in the project narrative.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of

that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery for Method 7470A, 70 – 130% for Method 245.1, and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as "amount" MSB. The Exception Code is changed to NC. The following two footnotes will appear on the report page—"NC: The recovery and/or RPD were not calculated.", and "MSB: The recovery and RPD were not calculated, because the sample amount was greater than four times the spike amount."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For Method 7470A, the ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument re-calibrated. (See Section 11.2.8 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include re-preparation of the ICV, ICB, CRA, CCV, and CCB with the calibration curve.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. For Method 245.1, the CCV must be 5% immediately following the calibration. All other CCVs must be 80 – 120%. A CCB is analyzed immediately following each CCV. (See Section 11.2.8 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. If the CCV/CCB is biased high and the sample results associated with the CCV/CCB are below the requested reporting limit then the results can be reported. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs.

9.9. Detection Limit Standard (CRA)-To verify linearity at the reporting limit, a CRA standard is run at the beginning of each sample analysis run after the ICV/ICB. The CRA standard mercury concentration is 0.2 ug/L. Recovery must be ± 50% of the true value, or the standard is either rerun or the problem corrected and the instrument re-calibrated. The CRA is only required when requested. (See Section 11.2.8 for the required run sequence.)

9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Section 11.2.9 for additional information on when full 4 point MSA is required as well as Appendix B for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.3. Set up the instrument with the operating parameters recommended by the manufacturer and listed in Appendix F. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.4. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the TestAmerica North Canton reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7 and Table I for additional information on preparing calibration standards and calibration levels.

10.5. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient. NOTE: If any digested standard does not meet criteria, all Ohio VAP samples associated with that curve will be re-digested.

10.6. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Sample Preparation:

11.1.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (7.3) into a series of 100 ml class A volumetrics, then dilute to volume . For the ICV, use a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards.

11.1.2 Transfer 100 mL of well-mixed sample or standard to a clean sample digestion bottle.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

Note: Spiking is done before the addition of acids or reagents.

11.1.3 Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 mixing after each addition.

11.1.4 Add 15 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 25-mL additional permanganate the color does not persist, sample dilution prior to reanalysis may be required.

Note: When performing analyses using automated vs. manual techniques the sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for by the addition of the same volume of permanganate to all other associated samples and standards in the run. In instances, where this is not feasible, the addition of excess reagent can be addressed through mathematical correction of the results to account for the resultant dilution effect.

11.1.5 Add 8 mL of potassium persulfate solution and heat for two hours in a water bath at 90 - 95 °C.

11.1.6 Cool samples.

11.2. Sample Analysis:

11.2.1 Refer to the Appendix F of this SOP for detailed setup and operation protocols.

11.2.2 When ready to begin analysis, add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains).

11.2.3 Automated determination: Follow instructions provided by instrument manufacturer.

11.2.4 Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.5 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.6 If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.7 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.8 The following run sequence is consistent with 7470A and 245.1.

Instrument Calibration
ICV
ICB
CRA
CCV
CCB
10 samples
CCV
CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for Quality Control criteria to apply to Methods 7470A and 245.1.

11.2.9 For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and matrix spike levels for TCLP analyses are detailed in Table I (Appendix A). Appendix B provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data are reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.5. Analytical Documentation

11.5.1 Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2 All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4 Sample results and associated QC are entered into the LIMs after final technical review.

11.6. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.7. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.6. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.7. Appropriate factors must be applied to sample values if dilutions are performed.

12.8. Sample results should be reported with up to three significant figures in accordance with the TestAmerica North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Method performance is determined by the analysis of method blanks, laboratory control samples, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

13.3.1 The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.3. Waste Streams Produced by the Method

15.3.1 **Acid Waste- Aqueous waste generated by the analysis.** Samples are disposed of in the acid waste drum located in the metals lab. The contents of the drum are neutralized and released to the POTW.

16. REFERENCES

16.1. References

16.1.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,

SW846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

16.1.2 "Methods for the Chemical Analysis of Water and Wastes", Rev. 3.0 (1994).

16.1.3 Corporate Quality Management Plan (QMP), current version.

16.1.4 TestAmerica Laboratory Quality Manual (LQM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1 QA Policy, QA-003.

16.2.2 Glassware Washing, NC-QA-0014.

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018.

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-0021.

16.2.5 Supplemental Practices for DoD Project Work, SOP, NC-QA-0016.

16.2.6 Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, Method 245.1 and CORP-MT-0006NC, current version.

16.2.7 Standards and Reagents, Sop NC-QA-0017.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1 Modifications from both 7470A and 245.1.

17.1.1.1 The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2 This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain

the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

17.1.2 Modifications from Method 7470A

17.1.2.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit if the samples associated with the method blank are equal to or above the reporting limit.

17.1.3 Modifications from 245.1

17.1.3.1 Method 245.1 states that standards are not heated. TestAmerica North Canton prepares heated standards for this method.

APPENDIX A

TABLES

TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (MG/L)

Standard Aqueous RL	0.0002
TCLP RL	0.002
Std 0	0
Std 1/CRA	0.0002
Std 2	0.0005
Std 3	0.001
Std 4	0.005
Std 5	0.010
ICV	0.0025
LCS/CCV	0.005
Aqueous MS	0.001
TCLP MS	0.005

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90-110 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.6).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero if the samples are at or above the reporting limit	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.6).
CRA	Beginning of every analytical run following the ICB and prior to sample analyses at or near the end of a run required only when requested	50-150% recovery	Rerun to verify; or correct problem and recalibrate or reprep with the calibration curve (See Sec. 9.8)
CCV	Every 10 samples and at the end of the run.	Recovery for Method 245.1 = 95-105% following a calibration. All other CCV for 245.1 are 80-120%. Recovery for Method 7470A = 80-120% following a calibration. If the CCV is biased high and the samples are < RL the results are acceptable.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve. (See Section 9.7).

CCB	Immediately following each CCV.	The result must be within +/- RL from zero. If the CCB is biased high and the samples are < RL the results are acceptable.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve. (See Section 9.7).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require re-digestion (See Section 9.3).	Re-digest and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. For Method 245.1, the LCS must be 85-115%.	Terminate analysis; Correct the problem; Re-digest and reanalyze all samples associated with the LCS (see Section 9.4).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.5) For TCLP see Section 11.2.9
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B

MSA GUIDANCE

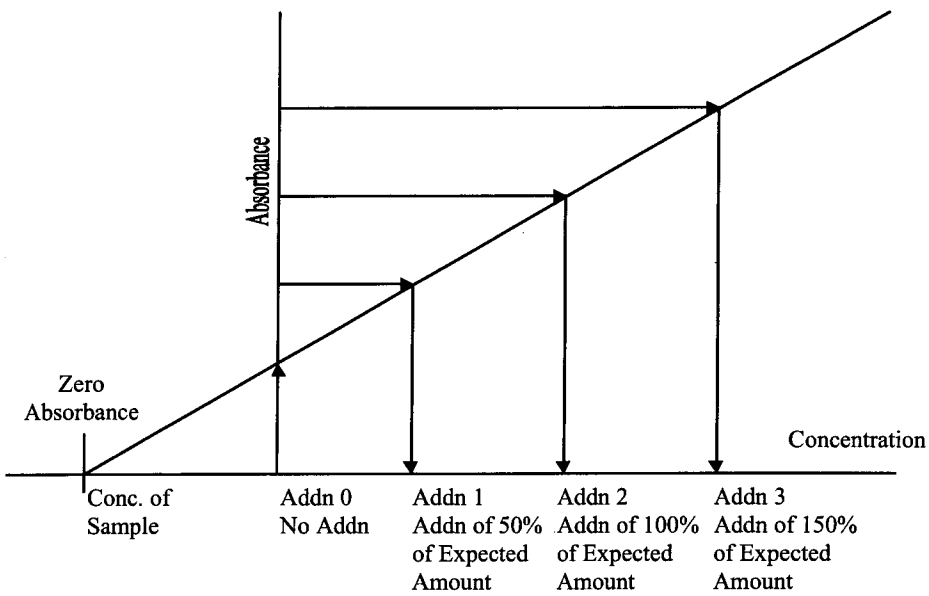
APPENDIX B. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX C

TROUBLESHOOTING GUIDE

APPENDIX C. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak Tubing blockage
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Lamp Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX D

CONTAMINATION CONTROL GUIDELINES

APPENDIX D. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Alternatively, vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX E

PREVENTIVE MAINTENANCE

APPENDIX E. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200II)

Daily	As Needed
Check nitrogen flow.	Check Hg lamp intensity.
Check tubing.	Clean lens.
Check drain.	Check aperture.
	Replace drying tube.
	Change Hg lamp.
	Check liquid/gas separator.

APPENDIX F

INSTRUMENT SET UP

Hg Analysis (Leeman PS200II)

SYSTEM INITIALIZATION AND WARM UP

1. F1 Menu

2. Instrument

a. TASKMASTER

b. #4 Wake System Up Enter

The warming up period takes approximately 10 minutes.

TO SET UP INSTRUMENT FOR ANALYSIS

1. F1 Menu

2. Autosampler

   A. Rack Entry

   B. Edit (ex. Rack 1), Enter

   C. Cup ID - Enter (clears sample #'s)

   D. Extended ID- type in matrix of sample (water or solid), Enter.

   E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.

   F. F3 Print Screen

3. Press F2 Macro key and type in analyst's first name - Enter

   A. Enter folder name - ex. HG10306, Enter. If folder does not exist, type Y - Enter.

   B. Type in - "Rack 1", "Rack 2" etc. , Enter.

   C. Type in : FROM CUP TO CUP

   Ex. = 1 30

Do the same for position 2 if needed. If not needed, you must press Enter 3 times to begin analysis.

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Copy No. _____
Implementation Date: 9-10-07

SOP No. CORP-IP-0002NC
Revision No. 2.6
Revision Date: 07/29/07
Page 1 of 21

TESTAMERICA NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF SOILS, SW846 METHOD 3050B

(SUPERSEDES: REVISION 2.5, DATED 12/02/04)

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICP/MS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICP/MS for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes and sediments.

1.4. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A representative 1 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP and ICP/MS (Ag, Sb, Sn) analysis. The digestates are then filtered and diluted to 100 mL.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

3.2. Total Metals: The concentration determined on an unfiltered sample following digestion. Note that this method is designed to determine the total *environmentally available* metals.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.3. Boron and silica from the glassware will grow into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. 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.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.9. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.10. Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.11. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, steam bath or other heating source capable of maintaining a temperature of 90-99°C

6.2. Calibrated thermometer that covers a temperature range of 0-200°C

6.3. Griffin beakers of assorted sizes or equivalent

6.4. Vapor recovery device (Watch glasses, ribbed or other device)

6.5. Whatman No. 41 filter paper or equivalent

6.6. Funnels or equivalent filtration apparatus

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 100 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repippetors or suitable reagent dispensers

6.11. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes, 100uL, 500uL, 1mL-5mL.

6.12. Class A volumetric flasks

6.13. pH indicator strips (pH range 0 - 6)

6.14. Plastic bottles, 120-150mL or equivalent

6.15. Boiling Stones – Ultra Pura PTFE or equivalent

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as

defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP LCS/MS spike solution: Prepare the ICP LCS/MS working spike solutions from custom stock standards to the final concentration listed in Table II. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months.

7.4. ICP/MS spike solution. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are custom made with the exception of Ag, Sb, and Sn, so that the final concentrations after spiking equals the concentrations listed in Table III. The ICP working solution (Section 7.3) is used for the Ag, Sb, and Sn spiking solution, which is also listed in Table III.

7.5. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.6. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.7. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.9. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.10. 30% Hydrogen peroxide (H_2O_2), reagent grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date

of collection to the date of analysis.

8.2. Soil samples do not require preservation with the exception of mercury, which must be stored at 4 °C ± 2 °C until time of preparation.

9. QUALITY CONTROL

Table IV (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements or verified as detailed in TestAmerica Corporate S-Q-003 and TestAmerica North Canton SOP NC-QA-0021. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the TestAmerica North Canton reporting limit. Criteria for DoD work is noted in SOP NC-QA-0016.

9.1.2. Initial Demonstration Study- this requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOPs (CORP-MT-0001)

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.4.1. The MB is prepared by weighing a 1 g aliquot of boiling chips. The MB is then processed as described in Section 11.10.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Tables II and III provide the details regarding the stock, working standards and final spike concentrations for ICP and ICP/MS. Refer to Section 7.4 for instructions on preparation of the aqueous LCS.

9.5.1. The LCS is prepared by spiking a 1g aliquot of boiling chips with 2 mL of the working LCS/MS spike solution (Section 7.3) for ICP and ICP/MS (Ag, Sb, Sn) analysis and 1 mL of the LCS/MS solution (Section 7.4) for the ICP/MS analysis. The LCS is then processed as described in Section 11.10.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Tables II through III provide the details regarding the stock, working standards and final matrix spike concentrations for ICP

and ICP/MS. Refer to Sections 7.4 for instructions on preparation of the working matrix spike solutions.

9.6.1. The soil matrix spike sample is prepared by spiking a 1 g aliquot of a sample with 2mL of the working LCS/MS spike solution (Section 7.3) for ICP analysis and 1 mL of the LCS/MS solution (Section 7.4) for ICP/MS analysis. The matrix spike sample is then processed as described in Section 11.10.

10. CALIBRATION AND STANDARDIZATION

10.1. Hotplate or hotblock temperature must be verified daily for each unit used, and must be recorded in a hotplate/hotblock temperature log. The hotplate temperature should be verified by measuring the temperature of a beaker of reagent water placed on each hotplate/hotblock. For block digestors, use a tube containing water.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. The heating procedures are carried out in a properly functioning hood.

11.4. All samples are to be checked out of Sample Control with an electronic chain of custody.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. An automatic label printing programs is used to reduce transcription errors (QuantIMS option).

11.6. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project administrator for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.7. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.8. In some cases, both ICP/MS and ICP digestates are required on each sample. It is recommended that both aliquots be weighed out and processed at the same time.

11.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.10. Preparation of Soils, Sediments and Sludges for Analysis by ICP and ICP/MS.

11.10.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

11.10.2. For each digestion procedure required (i.e., ICP or ICP/MS), weigh a 1g portion of solid and record the exact weight to the nearest 0.01g. A 2g sample size may also be used if needed to meet the reporting limits.

11.10.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.

11.10.4. Spike each of the MS and MSD aliquots with 2 mL of the working LCS/MS spiking solution (Section 7.3) for ICP analysis and 1 mL of the LCS/MS spiking solution for ICP/MS analysis.

11.10.5. Prepare a beaker, or equivalent container, for the method blank.

11.10.6. Prepare a beaker, or equivalent container, for the LCS. Add 2 mL of the working LCS/MS spiking solution (Section 7.3) for ICP analysis and 1 mL of the LCS/MS spiking solution (Section 7.4) for ICP/MS analysis.

11.10.7. Add 10 mL of 1:1 HNO_3 and mix the sample.

11.10.8. Heat sample to 95° ±4° C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be reprepared. Allow sample to cool, if necessary.

11.10.9. Add 5 mL of concentrated HNO_3.

11.10.10. Reflux at 95° ±4° C for 30 minutes. (Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.)

11.10.11. Allow the samples to cool, if necessary.

11.10.12. Add approximately 2 mL of reagent water and 1 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.10.13. Heat sample until effervescence subsides.

11.10.14. Allow the sample to cool, if necessary.

11.10.15. Continue adding 30% H_2O_2 in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

Note: Do not add more than a total of 10 mL of 30 % H_2O_2.

11.10.16. Continue heating at 95° ±4° C until the volume is reduced to approximately 5-10 mL. Alternatively the sample may be heated for two hours.

11.10.17. If the sample is being prepared for ICP or ICP/MS (Ag, Sb, Sn) analysis, add 10 mL of concentrated HCL and reflux for an additional 10 minutes (15 minutes for DoD projects) without boiling. This step is omitted for ICP/MS elements not requesting Ag, Sb, and/or Sn.

11.10.18. Allow the sample to cool.

11.10.19. Wash down beaker walls and vapor recovery device with reagent water.

11.10.20. Filter sample through Whatman 41 filter paper or equivalent into a graduated cylinder or equivalent or a pre-weighed bottle. Other measuring bottles (for example, Corning Snap Seals™) may be used if their accuracy is documented and is better than ± 2%. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

11.10.21. Dilute sample to 100 mL with reagent water. The sample is now ready for analysis.

12. DATA ANALYSIS AND CALCULATIONS

Not Applicable

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the

analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

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

15.2.1.1. Acidic waste containing nitric acid generated by the extraction. This waste is disposed of in a designated container labeled "Acid Waste".

15.2.1.2. Contaminated disposable materials utilized for the analysis. This waste is disposed of in a designated container labeled "Solid Waste".

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996. Method 3050B.

16.1.2. Corporate Quality Management Plan (QMP), current version.

16.1.3. TestAmerica Laboratory Quality Manual (LQM), current version.

16.1.4. TestAmerica Corporate Safety Manual, M-E-0001 and TestAmerica North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. CORP-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010B and Method 200.7.

16.2.2. NC-MT-0002, Inductively Coupled Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8.

16.2.3. QA-003, TestAmerica North Canton QC Program.

16.2.4. Glassware Washing, NC-QA-0014

16.2.5. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.6. Method Detection Limits and Instrument Detection Limits, S-Q-003 and NC-QA-0021

16.2.7. Supplemental Practices for DOD Project Work, NC-QA-0016

16.2.8. Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants, as defined in the determinative SOPs, are allowed up to two times the reporting limit in the blank.

17.2. Documentation and Record Management

The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst, matrix, prep type (ICP or ICP/MS)
- Sample ID; initial weight/volume and final weight/volume
- Standards Documentation (source, lot, prep date, volume added)
- Reagents

APPENDIX A

TABLES

TABLE I. Method 3050A Approved Analyte List

ELEMENT	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

TABLE II. ICP Soil Matrix Spike and LCS Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Soil MS/LCS Level * (mg/kg)
Aluminum	100	200
Antimony	25	50
Arsenic	100	200
Barium	100	200
Beryllium	2.5	5
Cadmium	2.5	5
Calcium	2500	5000
Chromium	10	20
Cobalt	25	50
Copper	12.5	25
Iron	50	100
Lead	25	50
Lithium	50	100
Magnesium	2500	5000
Manganese	25	50
Molybdenum	50	100
Nickel	25	50
Potassium	2500	5000
Selenium	100	200
Silver	2.5	5
Sodium	2500	5000
Strontium	50	100
Thallium	100	200
Vanadium	25	50
Zinc	25	50
Boron	50	100
Tin	100	200
Titanium	50	100

* Final soil spike concentration based on the addition of 2.0 mL working spike (7.3) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE III. ICP/MS Soil Matrix Spike and LCS Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Soil MS/LCS Level * (mg/kg)
Antimony **	25	50
Arsenic	10	10
Barium	10	10
Beryllium	10	10
Cadmium	10	10
Chromium	10	10
Cobalt	10	10
Copper	10	10
Lead	10	10
Manganese	10	10
Molybdenum	10	10
Nickel	10	10
Selenium	10	10
Silver **	2.5	5
Strontium	10	10
Thallium	10	10
Vanadium	10	10
Zinc	10	10
Boron	10	10
Tin **	1 00	200
Titanium	10	10
Zirconium	10	10

- Final soil spike concentration based on the addition of 1.0 mL working spike (7.4) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

** In order to reduce the amount of sample digestions, Ag, Sb, and Sn will be spiked at the same concentration as the ICP spike levels. A 10x dilution is recommended for the MS/MSD and LCS before analysis.

TABLE IV. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: NC-MT-0002 CORP-MT-0001	Redigest and reanalyze samples.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: NC-MT-0002 CORP-MT-0001	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: NC-MT-0002 CORP-MT-0001	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: NC-MT-0002 CORP-MT-0001	See Corrective Action for Matrix Spike.

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

Title: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

[Method: SW846 7471A]

Approvals (Signature/Date):

Technology Specialist	12-5-07 Date	Health & Safety Coordinator	12-6-07 Date
Quality Assurance Manager	12/6/07 Date	Laboratory Director	12/7/07 Date
Technical Director	12/6/07 Date		

This SOP was previously identified as SOP CORP-MT-0007NC, Rev 2.5, dated 11/22/04

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A.

1.2. The associated LIMs method code is O9.

1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.4. Method 7471A is applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, wastes, wipes and sludge-type materials. All matrices require sample preparation prior to analysis.

1.5. The TestAmerica North Canton reporting limit for mercury in solid matrices is 0.033 mg/kg based on a 0.6 g sample aliquot (wet weight).

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in hydrochloric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate which is used to breakdown organic mercury compounds also

eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that

are at elevated pH can react violently when acids are added.

5.3. 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.
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 Compounds	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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.7. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.8. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining temperature of 90- 95 °C).

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3. Peristaltic pump which can deliver 1 L/min air.

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5. Recorder or Printer.

6.2.6. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.

6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that acheive the same purpose are also acceptable (i.e., Gortex filter).

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. BOD bottles or equivalent

6.4. Nitrogen or argon gas supply, welding grade or equivalent

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes

6.6. Class A volumetric flasks

6.7. Top-loading balance, capable of reading up to two decimal places

6.8. Thermometer (capable of accurate readings at 95 °C)

6.9. Disposable cups or tubes

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm) mercury standards (in 10% HNO_3) are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. The calibration standards must be prepared fresh daily from the working standard (7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample preparation bottles and proceeding as specified in Section 11.1

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.5. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.8. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.10. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.11. Stannous chloride solution: Add 50 g of stannous chloride and 25 mL of concentrated HCl, and bring to a final volume of 500 mL with DI water.

Note: Stannous sulfate may be used in place of stannous chloride. Prepare the stannous sulfate solution according to the recommendations provided by the instrument manufacturer.

7.12. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.13. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.

8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7471A, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The result of the MDL determination must be below the TestAmerica North Canton reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count for determining the size of a preparation batch.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level). Note: For Ohio VAP projects, the result must be below the reporting limit or samples must be redigested.

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).
- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**
- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried

through the entire analytical procedure. If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 70-130% recovery must be applied.

- In the instance where the LCS recovery is greater than the upper control limit and the sample results are less than RL, the data may be reported. Such action must be addressed in the project narrative.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The LCSD recovery is evaluated using the same control limits as the LCS. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 70 - 130 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 and Section 11.2.11 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the ICV, ICB, CRA, CCV and CCB with the calibration curve.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 and 11.2.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. If the CCV/CCB is biased high and the sample results associated with the CCV/CCB are below the requested reporting limit, then the results can be reported. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs.

9.9. Detection Limit Standard (CRA)-To verify linearity at the reporting limit, a CRA standard is run at the beginning of each sample analysis run after the ICV/ICB. The CRA standard mercury concentration is 0.2 ug/L. Recovery must be ± 50% of the true value, or the standard is either rerun or the problem corrected and the instrument re-calibrated. The CRA is only required when requested. (See Section 11.2.8 for the required run sequence.)

9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the TestAmerica North Canton reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.4 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient. NOTE: If any digested standard does not meet SW846 criteria, all associated Ohio VAP samples must be redigested.

10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Standard and Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (7.3) into a series of sample digestion bottles. For the ICV, transfer a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3. Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under Section 11.1.5 below.

11.1.4. Transfer 0.6 g of a well mixed sample into a clean sample digestion bottle. Continue preparation as described under Section 11.1.5.

11.1.5. Water Bath protocol

11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.

11.1.5.2. To each **sample** bottle: Add *10* mL of reagent water and 5 mLof aqua regia.

11.1.5.3. Heat for two minutes in a water bath at 90 - 95 ° C.

11.1.5.4. Add *40* mL of distilled water.

11.1.5.5. Add 15 mL of potassium permanganate solution.

11.1.5.6. Heat for 30 minutes in the water bath at 90 - 95 °C.

11.1.5.7. Cool.

11.1.5.8. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.9. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add 50 mL of reagent water.

11.1.5.10. Continue as described under Section 11.2.

11.2. Sample Analysis

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. Manual determination:

11.2.3.1. Treating each sample individually, purge the headspace of the sample bottle for at least one minute.

11.2.3.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.

11.2.3.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.

11.2.3.4. Place the aeration device into 100 mLs of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.4. Automated determination: Refer to Appendix G for instrument setup and operation.

11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. micrograms (ug) of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.6. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.7. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.8. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.9. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.10. The following analytical sequence must be used with Method 7471A:

Instrument Calibration
ICV
ICB
Maximum 10 samples
CCV
CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Method 7471A.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.11. The following run sequence is consistent with Method 7471A and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
CRA
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

11.2.12. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) Recovery of the analyte in the matrix spike is not at least 50%,

2) The concentration of the analyte does not exceed the regulatory level, and,

3) The concentration of the analyte is within 20% of the regulatory level.

Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of

samples and standards, preventive maintenance and troubleshooting.

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.5. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C \times V \times D)/(W \times S)$$

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C)/(W \times S)$$

Where:

C = Concentration (ug) from instrument readout
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Sample results should be reported with up to three significant figures in accordance with the TestAmerica North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.1.

13.2. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 30 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.4. The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by this Method

15.2.1. The following waste streams are generated by this method.

15.2.1.1. Acid Waste. This waste disposed of in the designated container labeled "Acid Waste".

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

16.1.2. Corporate Quality Management Plan (QMP), current version.

16.1.3. TestAmerica Laboratory Quality Manual (LQM), current version. Associated SOPs and Policies, latest version

16.1.4. TestAmerica Corporate Safety Manual, M-E-0001 and TestAmerica North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version.

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021 and S-Q-003.

16.2.5. Supplemental Practice for DoD Project Work, SOP NC-QA-0016

16.2.6. Standards and Reagents, SOP NC-QA-0017.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from Method 7471A

17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination

with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7471A

17.1.2.1. Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Nonconformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Water Bath Procedure

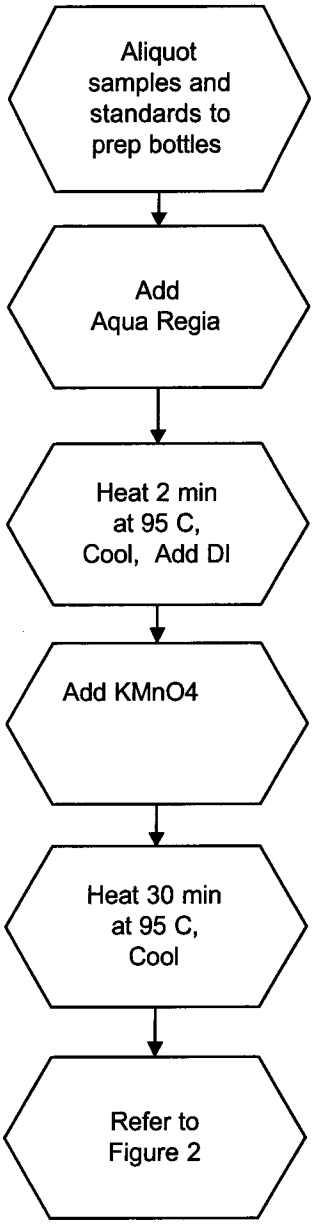
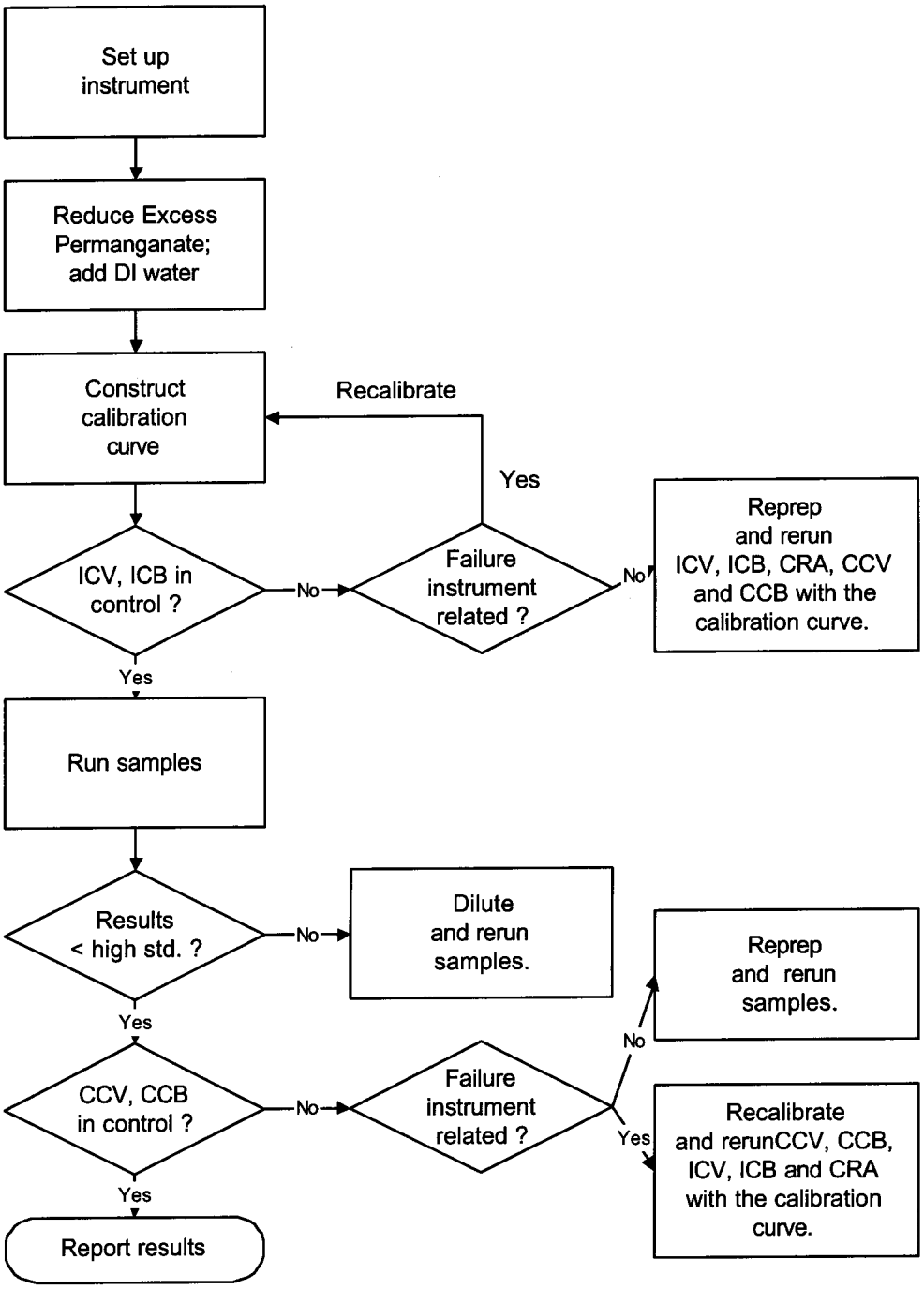


Figure 2. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Std 0 (mg/L)	0
Std 1 (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.005
Std 5 (mg/L) **	0.010
ICV (mg/L)	0.001 or 0.0025 ***
CCV/LCS/LCSD (mg/L)	0.0025 or 0.005 ***
MS (mg/L)	0.001

* SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.

** Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.

*** Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.

TABLE II. Summary Of Quality Control Requirements

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
ICV	Beginning of every analytical run	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve (see Section 9.7)
ICB	Beginning of every analytical run, immediately following the ICV	The result must be within +/- RL from zero	Terminate analysis, correct the problem, recalibrate or reprep with calibration curve (see Section 9.7)
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery	Terminate analysis, correct the problem, recalibrate and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve. If CCV is biased high and samples are ND, results can be reported (see Sections 9.8 and 10.6).
CCB	Immediately following each CCV	The result must be within +/- RL from zero	Terminate analysis, correct the problem, recalibrate and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve. If CCB is biased high and samples are ND, results can be reported (see Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples	The result must be less than or equal to the RL Sample results greater than 20x the blank concentration are acceptable Samples for which the contaminant is < RL do not require redigestion (see Section 9.4)	Redigest and reanalyze samples Note exceptions under criteria section See Section 9.4 for additional requirements

*See Sections 11.2.10 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Cont'd)

QC Parameter	Frequency *	Acceptance Criteria	Corrective Action
Laboratory Control Sample/Laboratory Control Sample Duplicate(LCS/LCSD)	One per sample preparation batch of up to 20 samples	Aqueous LCS/LCSD must be within 80 - 120% recovery or in-house control limits	Terminate analysis, correct the problem, redigest and reanalyze all samples associated with the LCS (see Section 9.5)
Matrix Spike	One per sample preparation batch of up to 20 samples	75 - 125 % recovery or in-house control limits If the MS/MSD is out for an analyte, it must be in control in the LCS	In the absence of client specific requirements, flag the data No flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.3.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits RPD ≤ 20% (see MS)	See Corrective Action for Matrix Spike

APPENDIX B

EXAMPLE
TESTAMERICA NORTH CANTON Hg DATA REVIEW CHECKLIST

Example
TestAmerica North Canton Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **7470A / 245.1 :** CORP-MT-0005 Rev 1 **7471 :** CORP-MT-0007 Rev 1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ **Date:** _____
Comments:
_____
_____

2nd Level Reviewer : _____ **Date:** _____
_____
_____

APPENDIX C

MSA GUIDANCE

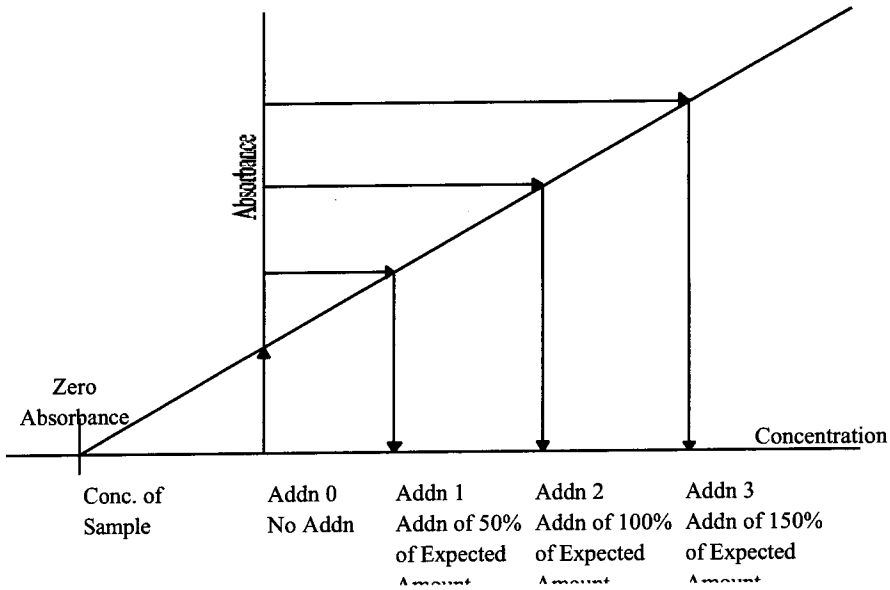
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) [1]

Daily	Semi-annually	Annually
Clean lens	Check Hg lamp intensity	Change Hg lamp
Check aperture		Check liquid/gas separator
Check argon flow		
Check tubing		
Check drain		
Replace drying tube		

Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	Monthly
Clean aspirator by flushing with DI water	Clean cell in aqua regia
Check tubing and replace if needed	Clean aspirator in aqua regia
Clean windows with methanol	
Change silica gel in drying tube	
Check argon gas supply	
Adjust lamp	

APPENDIX G

INSTRUMENT SET UP

Hg Analysis (Leeman PS200II)

SYSTEM INITIALIZATION AND WARM UP

1. F1 Menu
2. Instrument
   a. Taskmaster
   b. #4 Wake System Up - Enter

The warming up period takes approximately ten minutes.

TO SET UP INSTRUMENT FOR ANALYSIS

1. F1 Menu
2. Autosampler
   A. Rack Entry
   B. Edit (ex. Rack 1), Enter
   C. Cup ID - Enter (clears sample #'s)
   D. Extended ID- type in matrix of sample (water or solid), Enter.
   E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.
   F. F3 Print Screen
3. Press F2 Macro key and type in analyst's first name - Enter
   A. Enter folder name - ex. HG0306, Enter. If folder does not exist, type Y - Enter.
   B. Type in - "Rack 1", "Rack 2" etc. , Enter.
   C. Type in : FROM CUP TO CUP

      Ex. = 1 30

Do the same for position 2 if needed. If not needed, you must press "Enter" three times to begin analysis.

Title: ALKALINITY (TOTAL)

[Standard Method 2320B and EPA 310.1]

Approvals (Signature/Date):

Technology Specialist	2/21/08 Date	Health & Safety Coordinator	2-22-08 Date
Quality Assurance Manager	2/21/08 Date	Laboratory Director	2/22/08 Date

This SOP was previously identified as SOP NC-WC-0006, Rev 6, dated 01/30/06

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1. SCOPE AND APPLICATION

1.1. This method is applicable for the determination of total alkalinity in drinking, surface, saline, domestic, and industrial waters and wastewaters. It is also applicable to the determination of water-soluble alkalinity in solid samples if they have been prepared according to NC-IP-0009. It is based on EPA Method 310.1 and Standard Methods 2320B. The working linear range is 5 to 2500 mg/L.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

1.3. QuantIMs reference for total alkalinity is VC (310.1) and LV (2320B).

2. SUMMARY OF METHOD

2.1. An unaltered sample is titrated to an electrometrical endpoint of pH 4.5. **The sample must not be filtered, concentrated, or altered in any way.**

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Laboratory Quality Manual (QAM), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Samples with salts of weak organic and inorganic acids and greases or oils will interfere with pH measurements.

4.3. The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 mL.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. 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
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.			

5.3. Eye protection, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of an TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Alkalinity – Manual

6.1.1. Stir plate and stir bars

6.1.2. Graduated cylinders: various

6.1.3. Beakers: various

6.1.4. Buret: Class A 25 mL or 50 mL (preferred)

6.2. Alkalinity - Automated

6.2.1. Autotitrator

6.2.2. 50 mL centrifuge tubes

6.3. Alkalinity – Manual and Automated

6.3.1. pH meter and electrode(s) with temperature compensation

6.3.2. Volumetric pipettes: various

6.3.3. Autopipettor and disposable tips

6.3.4. Top loading balance: Capable of accurately weighing ±0.01 g

6.3.5. Volumetric flasks: various

6.3.6. Oven

6.3.7. Desiccator

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. 0.02 N Sulfuric Acid: reagent grade, purchased, standardized monthly.

7.1.2. Sodium Carbonate (Na_2CO_3): standard grade, dry overnight in 180°C oven, and cool in a desiccator or purchased primary standard grade.

7.1.3. Sodium Carbonate Solution: Add 2.50g of Na_2CO_3 (record exact weight of Na_2CO_3 used) to a 1000mL volumetric flask and dilute to volume with reagent water. Mix well.

7.2. Standards

7.2.1. Target Calibration Standard

7.2.1.1. pH Buffers: 4, 7, and 10 (manufactured)

7.2.2. Laboratory Control Sample

7.2.2.1. Alkalinity Standard, 25,000 mg/L $CaCO_3$, purchased or other commercially available reference solutions

7.2.3. Matrix Spike Standard

7.2.3.1. Alkalinity Standard, 25,000 mg/L $CaCO_3$, purchased

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers at 4°C ± 2°C.

8.3. The holding time is 14 days from sampling to analysis.

8.4. The bottle must be filled with no headspace and provided in a separate container.

8.5. Do not open sample bottle before analysis. If other tests are to be performed from the same bottle, Alkalinity must be determined first. This is dependent on the client sending a separate bottle for alkalinity.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24-hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. A reagent water blank consisting of 50 mL of reagent water and all other reagents added to samples within the analytical batch is analyzed with each analytical batch of samples.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. An LCS consisting of 1mL of the 25,000 mg/L alkalinity standard and 50 mL reagent water or other commercially available reference solution is analyzed with each analytical batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.3.3. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. An MS/MSD consisting of 1 mL of the 25,000 mg/L alkalinity standard and 50 mL of the sample will be analyzed.

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as DIL (diluted out).

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to laboratory limits.

9.4.3.4. If client program requirements specify to confirm matrix interferences, repreparation and reanalysis of the MS/MSD may be necessary.

9.5. QC Acceptance Criteria

9.5.1. Control limits are established by the laboratory as described in NC-QA-0018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOPs CA-Q-S-006 and NC-QA-0021.

9.6.2. MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument Directions

10.1.1. Calibrate the pH meter according to the manufacturer's specifications. See pH Electrode Method SOP # NC-WC-0010.

10.2. Initial Calibration

10.2.1. The pH meter is calibrated everyday with the 4 and the 7 calibration buffers and is verified at the beginning of the run by using the 10 buffer. The pH buffers should bracket the sample concentration.

10.3. Continuing Calibration

10.3.1. The pH meter is checked every ten readings with a midrange (pH 7) buffer to ensure the calibration remain linear. The acceptance range for the calibration check is 7 ± 0.05 pH units or recalibration is necessary.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be

completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. For solids preparation, see SOP NC-IP-0009.

11.3.2. No preparation is necessary for water samples.

11.4. Standardization

11.4.1. To standardize 0.02 N sulfuric acid, titrate 50 mL reagent water and 0.125 g sodium carbonate (weighed accurately and recorded) with 0.02 N H_2SO_4 to a pH of 4.5. This should be performed monthly or on a new lot of acid (whichever is more frequent). Calculate as follows:

$$N = \frac{A \times 1000}{53.00 \times B} \text{ (manual)}$$

$$N = \frac{A \times 1000}{53.00 \times B} \times \frac{20}{50} \text{ (Autotitrator)}$$

where: A=gNa_2CO_3

B=mL .02 N H_2SO_4 titrant

11.5. Repeat standardization two or three more times. Record standardization in the calibration logbook.

11.6. Sample Analysis – Manual

11.6.1. Do not shake sample.

11.6.2. Record the initial pH prior to sample analysis.

11.6.3. Use a sufficiently large volume of titrant (>20 mL in a 50 mL buret) to obtain good precision while keeping the volume low enough to permit a sharp end point

11.6.4. Place 50 mL of sample, or an aliquot diluted to 50 mL with reagent water, in a beaker. Begin mixing; measure and record initial pH of the sample. Titrate the sample to an endpoint of pH 4.5 with 0.02 N H_2SO_4. Record the volume of the titrant on the analytical logsheet. Samples requiring >50 mL titrant (> 75 mL using autotitrator) should be re-analyzed using less sample volume. Record dilution.

11.6.5. Use a sufficiently large volume of titrant (>20 mL in a 50 mL buret) to obtain good precision while keeping the volume low enough to permit a sharp end point.

11.7. Sample Analysis – Automated – Summary

11.7.1. The samples are analyzed on the autotitrator for Total Alkalinity.

11.7.2. Do not shake sample.

11.7.3. Place 50 mL of sample, or an aliquot diluted to 50 mL with reagent water, in a 50 mL centrifuge tube. See Manufacturer's information for operating instructions.

11.7.4. If a dilution of the sample was done change the volume on the schedule to reflect the dilution(based on a 20 mL sample inject) For alkalinity the dilution factor will be taken into account in the final calculation. Do not manually multiply the dilution unless it was not typed into the schedule.

11.7.5. After the results have been gathered from the instrument make sure to check the pH of all the samples. If a sample has an initial pH of >4.5 and the Total Alkalinity is zero, the sample must be diluted and reanalyzed.

11.8. Analytical Documentation

11.8.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.8.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. The supervisor or designee reviews logbooks.

11.8.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.8.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. *Alkalinity, mg/L $CaCO_3$ to pH 4.5* $= \dfrac{A \times N \times 50{,}000}{mL\ of\ sample}$

12.2. LCS % $= \dfrac{mg/L}{500\ (true)} \times 100$

MS/MSD % $= \dfrac{B - C}{500\ (true)} \times 100$

where:

A = mL of Titrant
N = Normality of Titrant
B = MS/MSD, mg/L
C = Sample, mg/L

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that an associate who has been properly trained in its use and has the required experience performs this procedure.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and

the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

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

15.2.1.1. This waste is drained from the titration cell into the aqueous waste stream since the pH range is between 4 and 10. Any sample waste generated that is not in this pH range is collected in a designated container identified as "Acid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica North Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. EPA-600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Alkalinity, Method 310.1.

16.1.2. Standard Methods for the Examination of Water and Wastewater, 18th Edition, 1992,Alkalinity Methods, 2320B.

16.1.3. EPA 600, Methods for Chemical Analysis of Water and Wastes, pH, Method 150.1.

16.1.4. TestAmerica North Canton Laboratory Quality Manual (QAM), current version.

16.1.5. TestAmerica Corporate Safety Manual, M-E-0001 and TestAmerica North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs

16.2.1. Solid Extraction for Wet Chemistry Parameters, NC-IP-0009.

16.2.2. pH Electrode Method for Wet Chemistry Parameters, NC-WC-0010.

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018.

16.2.4. QA Policy, QA-003

16.2.5. Glassware Washing, NC-QA-0014

16.2.6. Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and NC-QA-0021

16.2.7. Navy/Army SOP, NC-QA-0016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The lower reporting limit (RL) for undiluted samples is 5 mg/L $CaCO_3$.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method Deviation

17.2.1. A fixed endpoint of 4.5 is used for all samples since the sample concentration is often unknown.

17.2.2. The Sodium Carbonate (Na_2CO_3) is dried at 180°C overnight instead of at 250° C for 4 hours.

17.2.3. The standard acid solution is not boiled gently for 3-5 minutes under a watch glass cover.

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. NC-WC-0010, Rev. 7
Effective Date: 03/23/06
Cover Page

Title: pH Electrometric Method
[Methods: SW9040B, SW9045C, EPA150.1]

Approvals (Signature/Date):

Technology Specialist	12/14/07 Date	Health & Safety Coordinator	12-14-07 Date
Quality Assurance Manager	12/13/07 Date	Laboratory Director	12/14/07 Date

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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: PH ELECTROMETRIC METHOD

(SUPERSEDES: REVISION 6, DATED 10/27/04)

Reviewed by:	Technology Specialist	6/8/06 Date
Approved by:	Quality Assurance Manager	5/16/06 Date
Approved by:	Environmental Health and Safety Coordinator	3-27-06 Date
Approved by:	Laboratory Director	6/13/06 Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of pH in waters, wastewaters, and solids. It is based on SW846 Methods 9040B and 9045C and EPA Method 150.1. The approximate working range is 1 - 14 pH units. Samples with a pH of < 1 are reported as < 1.

1.2. The associated method codes are PU (9040B), OZ (9045C), and AJ (150.1). The preparation codes are 88 and 1C.

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. The pH is determined electrometrically by using an electrode. The pH meter is calibrated with a series of known pH buffers.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. 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.

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves

that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents {as well as glassware cleaning procedures that involved solvents such as methylene chloride} should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of an STL North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. pH meter with electrode(s) and temperature compensation

6.2. Beakers: various

6.3. Top loading balance: Capable of accurately weighing ±0.01 g

6.4. Stir plate and stir bars

6.5. Shaker or mechanical tumbler

6.6. Autotitrator

6.7. Centrifuges tubes

7. REAGENTS AND STANDARDS

7.1. Standards

7.1.1. Target Calibration Standards

7.1.1.1. pH 2, 4, 7, 10, and 12 buffers, purchased

7.1.1.2.Fresh buffers are poured and used each working day.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers at 4°C ± 2°C.

8.3. Samples should be analyzed as soon as possible after sampling, but not to exceed twenty-four hours.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS and Sample Duplicate) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Sample Duplicate

9.2.1. A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.2.2. Sample duplicates are performed at a frequency of 10% per matrix, and must meet laboratory-specific limits for precision.

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each analytical batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A commercially available (Environmental Resource Associates or equivalent) control standard will be analyzed. Recovery must be within +/- 2% of true value.

9.3.3. Corrective Action for LCS

9.3.3.1. If the pH is outside the established control limits the system is out of control and corrective action must occur.

9.3.3.2. Corrective action consists of identification and correction of the cause for the out of control situation and reanalysis of all effected samples.

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1. Refer to the manufacturer's manual for instrumental calibration.

10.1.2. The following procedure is applicable for use with the Orion 250 pH meter.

10.1.2.1. Rinse the electrodes with reagent water and place in the pH 4.0 buffer. Press "Cal". Allow the value to stabilize and then, using the arrow keys, adjust the value up or down to read 4.00. Press Enter.

10.1.2.2. Rinse the electrodes and place in the pH 7.0 buffer. Allow the value to stabilize and then, using the arrow keys, adjust the value up or down to read 7.00. Press Enter.

10.1.2.3.Calibration Check: Rinse the electrodes and place in the pH 10.0 buffer. Allow value to stabilize. The pH should be between 9.95 and 10.05 or recalibration is necessary.

NOTE: When analyzing drinking water samples, calibrate as described in section 10.1.2, using the pH 7.0 and pH 10.0 buffers for calibration and the pH 4.0 buffer for the calibration check.

10.1.3The pH meter should be calibrated daily. The calibration is recorded on the analytical logsheet.

10.1.4If the pH meter has been turned off, it must be calibrated prior to use.

10.2. Continuing Calibration

10.2.1. A pH 7 buffer is analyzed before analysis and every ten samples to ensure the calibration remains linear.

10.2.2. The pH meter must be recalibrated if the buffer deviates by more than± 2%. If this range is exceeded, reanalyze all samples analyzed since the last pH buffer that met criteria.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Waters

11.3.1.1.No preparation necessary for waters and wastewaters.

11.3.2. Solids and Soils

11.3.2.1.Place 20 g (± 0.5 g) of sample in a beaker or other suitable container.

11.3.2.2.Add 20 mL of reagent water and mix for five minutes.

11.3.2.3.Allow sample to stand for one hour to allow the solids to settle out.

11.4. Sample Analysis

11.4.1. Manual Procedure

11.4.1.1.Waters

11.4.1.1.1.Place the sample in a clean beaker using a sufficient volume to cover the sensing elements of the electrode(s). Allow the pH to stabilize (swirling or stirring may quicken stabilization). Record the pH on the analytical logsheet. Remove the electrodes from the sample. Rinse and gently dab off the electrodes between each measurement. Store the electrodes in pH 7 buffer when not in use.

11.4.1.1.2. For 9040B – Continuously stir the sample while obtaining a stable reading.

11.4.1.2.Solids

11.4.1.2.1.Immerse the pH electrodes in the supernatant layer of the sample - be careful not to stir up solids. Allow pH to stabilize and record it on the analytical logsheet. Remove and rinse the electrodes between each measurement. Store electrodes in the pH 7.0 buffer.

NOTE: If the sample contains oil or other substances that will coat or damage the electrodes, the pH should be analyzed following SOP# NC-WC-0009, pH - Paper Method.

11.4.2. Automated Procedure

11.4.2.1.Load the appropriate schedule on the autotitrator, starting with the pH calibration.

11.4.2.2. Pour a homogenized sample into the centrifuge tubes and place the tubes in the appropriate position on the autosampler. Remember to include a pH 7 buffer check after every ten positions.

11.4.2.3. Start the autotitrator.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not Applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

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

15.2.1.1. Acidic and alkaline sample waste and exhausted buffer solutions poured down the drain if the pH is between 4 and 10. Any sample waste generated that is not in this pH range is collected in a designated container identified as "Acid Waste".

15.2.1.2. Exhausted soil or oil samples analyzed by the method. The liquid layer is decanted and disposed of in a designated container identified as "Acid Waste". The remaining solid layer is disposed of by placing it in a container identified as "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL North Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, 3rd Edition, pH Electrometric Measurement, Method 9040B

16.1.2. EPA 600, Methods for Chemical Analysis of Water and Wastes, pH (Electrometric), Method 150.1

16.1.3. SW846, Test Methods for Evaluating Solid Waste, 3rd Edition, Soil pH, method 9045C.

16.1.4. STL North Canton Laboratory Quality Manual (LQM), current version

16.1.5. Corporate Quality Management Plan (QMP), current version.

16.1.6. STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5. Navy/Army SOP, NC-QA-0016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. A minimum reporting limit is not listed in LIMS. Units are reported as No Units.

TESTAMERICA North Canton
STANDARD OPERATING PROCEDURE

TITLE: TOTAL ORGANIC CARBON (TOC)

(SUPERSEDES: REVISION 2.3, DATED 12/16/04)

Reviewed by:	Technology Specialist	9/7/07 Date
Approved by:	Quality Assurance Manager	10/4/07 Date
Approved by:	Environmental Health and Safety Coordinator	10-1-07 Date
Approved by:	Laboratory Director	10/3/07 Date

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1. SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Total Organic Carbon (TOC) in waters and similar matrices. It is based on SW846 Method 9060 and EPA Method 415.1. The working linear range is instrument dependent at 1 mg/L to 50 mg/L with a reporting limit of 1 mg/L.

1.2 QuantIMS method codes are DA (415.1) and FM (9060).

1.3 This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1 Organic Carbon is converted to carbon dioxide (CO_2) using chemical oxidation. The CO_2 is then measured by an infrared detector. The samples are purged prior to analysis such that only non-purgable organic carbon is being measured.

3. DEFINITIONS

3.1 Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause Method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Carbonate and bicarbonate interfere but are eliminated by the acidification and purging step of the instrument.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2 The auto sampler has a probe that is sharp; use caution not to stick yourself.

5.3 The furnace is very hot and can cause severe burns if touched.

5.4 The Sodium Persulfate is a strong oxidizer. Avoid contact with combustible materials, organic materials, strong reducing agents, and excess heat.

5.5 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
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	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.
Phosphoric Acid	Corrosive	1 Mg/M^3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Persulfate	Oxidizer Corrosive	0.1 Mg/M^3- TWA as Persulfates	Causes irritation to the respiratory tract. Symptoms may include sore throat, shortness of breath, inflammation of nasal passages, coughing, and wheezing. Causes severe irritation or burns to the skin and eyes. Symptoms include redness, itching, pain and burns. May cause allergic skin reactions. Can cause eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.6 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut.

Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.7 Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.8 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.9 It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.10 Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.11 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1 O·I Corporation Model 1010 TOC Analyzer with 1051 vial multisampler

6.2 Nitrogen Gas and Regulator

6.3 Volumetric flasks: Various sizes

6.4 Volumetric pipettes: Various sizes

6.5 Vials: 40 mL glass

6.6 Graduated cylinders: Various sizes

6.7 pH Strips

6.8 Whatman filter #4

6.9 Top loading balance: capable of accurately weighing ± 0.01 g

7. REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Sodium Persulfate: Reagent Grade

7.1.2 Sodium Persulfate Solution: Add 200 g sodium persulfate ($Na_2S_2O_8$) to a 1 liter volumetric flask and dilute to volume with reagent water.

7.1.3 Phosphoric Acid, concentrated: Reagent Grade

7.1.4 Phosphoric Acid Solution: Carefully add 59 mL concentrated phosphoric acid (H_2PO_4) to 900 mL of reagent water in a 1 liter volumetric flask. Dilute to volume with reagent water.

7.1.5 Sulfuric Acid, concentrated: Reagent Grade

7.2 Standards

7.2.1. All standards should be prepared in volumetric flasks, using volumetric pipettes, and diluted to volume with reagent water.

7.2.2. TOC Stock Standard

7.2.3. Primary and secondary sources are needed.

7.2.3.1.TOC 1000 mg/L

7.2.3.1.1. Dilute 1.06 g KHP (potassium acid phthalate) to volume in a 500 mL volumetric flask. A commercially prepared solution may also be used.

7.2.3.2.Prepare every six months.

7.2.4. TOC Calibration Standards

7.2.5. Prepare the following standards from the primary stock standard described in Section 7.2.3.1.1.

Concentration (mg/L)	Volume (mL)	Stock Concentration (mg/L)	Final Volume (mL)
50	5	1000	100
25 (CCV/MS/MSD)	6.25	1000	250
10	1	1000	100
1	0.1	1000	100

7.2.6. TOC Verification Standard (LCS)

7.2.6.1. A commercially prepared solution is used.

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples are preserved to a pH <2 with sulfuric acid (H_2SO_4) or hydrochloric acid (HC1) and stored in plastic or glass containers at 4°C±2°C.

8.2 The holding time is 28 days from sampling to analysis.

9 QUALITY CONTROL

9.1 Batch Definition

9.1.1 A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2 Method Blank

9.2.1 One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to

the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2 A method blank consisting of 40 mL of reagent water and all reagents added to the samples must be prepared and analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.2.3 Corrective Action for Blanks

9.2.3.1 If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2 If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3 Laboratory Control Sample (LCS)

9.3.1 Laboratory Control Samples are well characterized; laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.3.2 A purchased LCS must be analyzed with each batch of samples.

9.3.3 Corrective Action for LCS

9.3.3.1 If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2 The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.3.3 Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1 One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2 An MS/MSD consisting of 20 mL of sample and 20 mL of the 25 mg/L standard will be analyzed with each analytical batch of samples.

9.4.3 Corrective action for MS/MSDs

9.4.3.1 If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2 If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as "amount" MSB. The Exception Code is changed to NC. The following two footnotes will appear on the report page 'NC The recovery and/or RPD were not calculated." "MSB The recovery and RPD were not calculated because the sample amount was greater than four times the spike amount."

9.4.3.3 If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.5 Control Limits

9.5.1 Control limits are established by the laboratory as described in SOP NC-QA-0018.

9.5.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via the LIMs (QC Browser program).

9.6 Method Detection Limits (MDLs) and MDL Checks

9.6.1 MDLs and MDL Checks are established by the laboratory as described in SOP, NC-QA-0021 and S-Q-003.

9.6.2 MDLs are easily accessible via the LIMs (QC Browser program).

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10 CALIBRATION AND STANDARDIZATION

10.1 Recommended Initial Setup

Constant Settings		
STD Mass	=	6.76 ug C
Sample Vol	=	2.0 mL
Acid Vol	=	4 x 100 uL
Oxidant Vol	=	10 x 100 uL

10.1.1 Adjust the nitrogen to 30 psi using the flow valve on the tank. The gauge should always be set at 30 psi when the instrument is not in use.

10.1.2 Remove the reagent bottles and fill with appropriate reagents (phosphoric acid solution and sodium persulfate). Do not fill bottles completely full; leave a small amount of air space. Loosely reconnect caps (and tubing), and replace the bottles into the instrument.

10.1.3 Blank and calibrate the instrument when CCVs and/or CCBs fail to meet acceptance criteria or when other problems are encountered.

10.1.3.1. Choose "Calibration". Start a new file with the current date.

10.1.3.2. Choose "Sequences" from the "databases" menu option, open up the calibration template.

10.1.3.3. Confirm all information. If blanking is required, it is best if done before calibration. Enter the desired number of blanks (no less than five) in the "reagent blanks before" field.

10.1.3.4. Analyze an ICV/ICB

10.1.3.5. Save the file using the current date as the filename.

10.1.3.6. Update data file information.

10.1.3.6.1. Choose the setup menu option, go into win TOC output, change the log fill name and prefix counter.

10.1.3.7. When analysis is complete, print the run from "/utilities/view run log"

10.1.3.8. Evaluate the data. The correlation coefficient of the original curve must be =0.995 or recalibration is required.

10.2. Continuing Calibration

10.2.1 The run is checked at the beginning, after every ten samples, and at the end of the run of the same species using a midrange CCV made from a primary source (Section 7.2.4. or 7.2.7.) to verify continued linearity. A CCV cannot vary from the original curve by more than ± 10%, or recalibration is required.

10.2.2 System cleanliness is checked every ten samples and at the end of the run using Continuing Calibration Blank (CCB). A CCB cannot contain the analyte of interest above the reporting limit, or recalibration is required.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2. Sample Preparation Procedure

11.2.1. If excess particulate matter exists, filter an aliquot of sample through a Whatman #4 filter into a TOC vial or decant.

11.3. Sample Analysis Procedure

11.3.1 Type a run protocol sequence into the computer using the run template, if desired. Update the data file information in the setup/win TOC output.

11.3.2 For Method 9060, quadruplicate analysis is required. If quadruplicate reporting is requested, each of four results is reported. Replicate analysis should be taken from separate vials, if available. If only one reportable result is requested per sample, the four results should be taken from one vial, and the average of the four results are reported.

11.3.3 For Method 415.1 only one analysis is required. The single analysis is reported directly from the instrument printout.

11.3.4 All samples and standards should be poured into 40 mL vials. Samples received in vials can be run in those containers, provided there is not an excess of solids.

11.3.5 Be sure the samples are loaded on the sampler, the first one positioned under the needle.

11.3.6 Click the "Start" button.

11.3.7 Samples that fall outside the linear range (>50 mg/L) of the instrument must be diluted and reanalyzed.

11.3.7.1 Samples following a high sample should be re-analyzed if carryover is a concern.

11.3.8 Print the run from "utilities/view run log".

11.3.9 When analysis is complete, properly dispose of or put away samples and standards.

12. DATA ANALYSIS AND CALCULATIONS

12.1 Preparation Documentation

12.1.1 Record any sample preparation on the analytical logsheet.

12.2 Analytical Documentation

12.2.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

12.2.2 All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. The supervisor or designee reviews logbooks.

12.2.3 Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

12.2.4 Sample results and associated QC are entered into the Laboratory Information Management System (LIMS) after final technical review.

12.2.5 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12.3 Calculations for 9060 only

12.3.1 Total Organic Carbon, mg/L = Average Instrument Value x Dilution

Where:

TOC, mg/L = average of the 4 instrument values x dilution, calculated (without dilution) by the instrument.*

12.3.2 *LCS % Recovery* =

$$\frac{\textit{Iinstrument values}^{*}}{\textit{True Value}} \times 100$$

12.3.3 MS/MSD % recovery

$$\left(\frac{(\textit{Iinstrument values}^{*}\ \textit{MS or MSD}) - (\textit{Avg sample instrument value}^{*} \div 2)}{12.5}\right) x100$$

* One of the values may be judged erroneous and disregarded if three of the four are consistent. If no consistency can be found in the four values, the sample must be rerun.

13. METHOD PERFORMANCE

13.1 Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

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

15.2.1.1. Acidic waste from the auto-analyzer. This waste is disposed of in a designated container identified as "Acid Waste."

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total Organic Carbon, Method 9060.

16.1.2. EPA 600, Methods for Chemical Analysis of Water and Wastes, Organic Carbon, Method 415.1.

16.1.3. Corporate Quality Management Plan (QMP), current version.

16.1.4. TestAmerica Laboratory Quality Manual Plan (LQMP), current version.

16.1.5. TestAmerica Corporate Safety Manual, M-E-0001 and TestAmerica North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits, NC-QA-0021 and S-Q-003

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.6. Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The lower reporting limit is 1 mg/L

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide

17.2.1. See the manufacturer's instructions for an instrument troubleshooting guide and maintenance requirements.

17.3. Method deviations

17.3.1. A blender is not used to homogenize samples.

17.3.2. For Method 9060, the calibration must be verified with an independently prepared check standard every 15 samples. The laboratory is verifying the calibration every ten samples.

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. NC-WC-0032, Rev. 8.4
Effective Date: 12/08/04
Cover Page

Title: Cyanide, Preparation Method
[Methods: SW9012A, EPA335.1, 335.2, SM4500CN-I, SM4500CN-E, CLP ILM03.0]

Approvals (Signature/Date):

Technology Specialist	Date 12/14/07	Health & Safety Coordinator	Date 12-14-07
Quality Assurance Manager	Date 12/13/07	Laboratory Director	Date 12/14/07

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SOP No.: NC-WC-0032
Revision No.: 8.4
Revision Date: 12/08/04
Page 1 of 26

STL STANDARD OPERATING PROCEDURE

TITLE: CYANIDE PREPARATION METHOD

(SUPERSEDES: REVISION 8.3, DATED 06/17/03)

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Free Cyanide in solids, liquids, and waters. It is based on CLP ILM03.3, CLP ILM04.0, SW 846 Method 9012A, EPA 335.1, 335.2, 335.4, Standard Method 4500-CN-I and ASTM D 4282-83. The working linear range is 0.005 - 0.2 mg/L for waters and 0.25 to 10 mg/kg for solids.

1.2. The associated method codes are CLP ILM03.0 (DV), CLP ILM04.0 (OU), SW846 Method 9012A (QP), EPA 335.1 and 335.2 (CG), EPA 335.4 (O2), Standard Method 4500-CN-E (A4), Free Cyanide 4500CN-I (HF) and Amenable Cyanide 9012A (N4).

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Oxyidizing agents such as chlorine will decompose most cyanides. Sulfide will distill over with the cyanide and could affect colorimetric, titrimetirc, and electrode procedures. Refer to the preparation section on how to screen and treat samples appropriately.

4.3. Aldehydes convert cyanide to cyanohydrin which could result in the lost of cyanide. If the presence of aldehydes are suspected, stabilize the sample with NaOH at the time of collection and add 2 mL 3.5% ethylenediamine solution per 100 mL of sample.

4.4. Refer to Standard Methods for further information on possible interferences and associated treatments.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual the Waste Management SOP, and this document.

5.2. In the event the sample begins to react unexpectedly during distillation, remove entire apparatus from heat source, set aside, and allow to cool. DO NOT ATTEMPT TO DISASSEMBLE GLASSWARE. Doing so may result in a sudden release of pressure with spraying of the sample.

5.3. Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.4. Latex, vinyl, Nitrile or similar gloves may be used.

5.5. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.6. The acidification of samples prior to extraction/preparation can result in the release of a highly toxic gas, hydrogen cyanide.

5.7. If samples are identified with cyanide concentrations equal to or greater than 200 mg/L, immediately notify the department manager and personnel responsible for hazardous waste shipping. Those samples must be identified as extremely hazardous for other chemists and must receive special attention during disposal.**Potassium cyanide and sodium cyanide will give off Hyrdogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.**

5.8. Cyanide and cyanide salts are extremely toxic. Addition of acid can generate Hydrogen Cyanide gas, which can be extremely dangerous.

5.9. 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
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Acetic Acid (1)	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Bismuth Nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.

Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 Mg/M3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. 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.
Potassium Phosphate	Flammable	None	Inhalation causes severe irritation to the respiratory tract. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. .
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.
Lead Carbonate	Poison Neurotoxin Irritant Probable carcinogen Reproduc-tive hazard	0.05mg/m3 TWA as Lead	Inhalation can cause local irritation of bronchia and lungs and can cause symptoms such as metallic taste in the mouth, chest and abdominal pain. Skin contact can cause local irritation, redness and pain. Can be absorbed through the 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.
Phosphoric Acid	Corrosive	1 mg/m^3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m^3 – Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, swelling. Greater exposures cause severe burns with possible blindness.
Sodium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN (skin)	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.

Chloramine T Hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid contact and inhalation.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
Silver Nitrate	Corrosive Poison Oxidizer	$0.01 mg/m^3$ (TWA) for silver metal dust and fume as 0.02 Ag	This is a corrosive, poisonous material. It will cause burns to any area of contact and is harmful if inhaled. Ingestion may cause death. Contact with other material may cause fire. Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.10 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.11 Exposure to chemicals must be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. All samples with pink stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.12 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.13 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Cyanide Distillation Apparatus

6.2. Analytical balance, capable of accurately weighing ± 0.0001 g

6.3. Vacuum pump

6.4. Graduated cylinders: various

6.5. Volumetric flasks: various

6.6. Volumetric pipets: various

6.7. Balance: Top loading, capable of accurately weighing ± 0.01 g

6.8. Lead Acetate Indicator Paper

6.9. Potassium Iodide (KI) Indicator Paper

6.10. Erlenmeyer flasks: various

6.11. Buret: Class A 10 mL

6.12. pH strips

6.13. Boiling stones or chips

6.14. Beakers: various

6.15. Snap seal containers: 120 mL

6.16. Plastic bottles with lids: 250mL or 500mL

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfamic Acid: reagent grade (not used for CLP ILM03.0)

7.1.2. Sulfamic Acid Solution: Add 100 g of sulfamic acid to 800 mL reagent water and dilute to 1 liter with reagent water (not used for CLP ILM03.0)

7.1.3. Ascorbic Acid: reagent grade

7.1.4. Sodium Hydroxide: (NaOH), high purity grade.

7.1.5. Sodium Hydroxide, 1.25 N: Add 50 g of NaOH to 900 mL and dilute to 1 liter with reagent water.

7.1.6. Sodium Hydroxide, 0.25 N: Add 10 g of NaOH to 900 mL reagent water and dilute to 1 liter with reagent water. Purchased reagent may also be used.

7.1.7. Sulfuric Acid: (H_2SO_4), concentrated

7.1.8. Acetic Acid Solution: Add 10 mL of glacial acetic acid to 90 mL of reagent water.

7.1.9. Magnesium Chloride: ($MgCl_2 \bullet 6H_2O$), reagent grade

7.1.10. Magnesium Chloride Solution: Add 510 g $MgCl_2 \bullet 6H_2O$ to 500 mL reagent water and dilute to 1 liter with reagent water. Purchased reagent may also be used.

7.1.11. Calcium Hypochlorite: [Ca $(OC1)_2$], reagent grade

7.1.12. Calcium Hypochlorite Solution: Add 5 g of $Ca(OC1)_2$ to 100 mL of reagent water.

7.1.13. Methyl Red Indicator: Add 0.05 g of methyl red to 50 mL of glacial acetic acid and dilute to 100 mL with reagent water.

7.1.14. Methyl red reagent grade glacial

7.1.15. Acetic Acid: (CH_3COOH), glacial reagent grade

7.1.16. Zinc Acetate: [$Zn(C_2H_3O_2)$], reagent grade

7.1.17. Zinc Acetate Solution: Add 100 g zinc acetate to 800 mL reagent water and dilute to 1 liter with reagent water.

7.1.18. Sodium Acetate: [$NaC_2H_3O_2 \bullet 3H_2O$] reagent grade

7.1.19. Sodium Acetate Buffer: Add 410 g of sodium acetate to 500 mL of reagent water. Adjust the pH to 4.5 using glacial acetic acid and dilute to 1 liter with reagent water.

7.1.20. Rhodanine: reagent grade

7.1.21. 0.0192 N Silver Nitrate: reagent grade

7.1.22. Bismuth nitrate [$Bi(NO_3)_3$]: Dissolve 30 g of $Bi(NO_3)_3$ in 100 mL of reagent water. While stirring, add 250 mL of glacial acetic acid. Stir until dissolved. Dilute to 1 L with reagent water.

7.1.23. Cadmium chloride [$CdCl_2$] – anhydrous : Dissolve 1.0 g $CdCl_2$ in 90 mL of reagent water. Dilute to 100 mL with reagent water to produce a 10 g/L $CdCl_2$ solution.

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months.

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See SOP NC-WC-0031 Appendix I

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow 7.2.1 using an alternate source of Potassium Cyanide (KCN).

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See SOP NC-WC-0031 Appendix I.

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1. If using the 1.25 N NaOH: pipet the appropriate amount of cyanide standard into 100 mL volumetric and add 20 mL 1.25N NaOH to each calibration standard (except the 1.0 mg/L) and bring to volume with reagent water.

If using the purchased 0.25N NaOH: pipet the appropriate amount of cyanide standard into 100 mL volumetric and bring to volume with 0.25N NaOH. Prepare weekly.

Concentration CN-	mL CN-	Final Volume
100 mg/L	10 mL of 1000 mg/L	100 mL
10 mg/L	10 mL of 100 mg/L	100 mL
1.0 mg/L	10 mL of 10 mg/L	100 mL
*0.2 mg/L	20 mL of 1 mg/L	100 mL
*0.1 mg/L	10 mL of 1 mg/L	100 mL
*0.05 mg/L	5 mL of 1 mg/L	100 mL
*0.025 mg/L	25 mL of 0.1 mg/L	100 mL
*0.01 mg/L	10 mL of 0.1 mg/L	100 mL
*0.005 mg/L	10 mL of 0.05 mg/L	100 mL

*Denotes calibration standards

8. SAMPLE PRESERVATION AND STORAGE

8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH>12. All samples are stored at 4° C ± 2°C in plastic or glass containers.

8.2. The holding time for **non-CLP** samples is fourteen days from sampling to analysis.

8.3. The holding time for **CLP** samples holding time is twelve days from receipt to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. **Non-CLP**

A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same process.

9.1.2. **CLP**:

A batch is a group of no greater than 20 samples excluding QC samples (LCS/ICV, Method Blank, Sample Duplicate and Matrix Spike) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank (MB)

9.2.1. **Non-CLP**

One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.1.1. A method blank consists of 50 mL 0.25N NaOH for Total Cyanide analysis or 50 mL reagent water for Free and Amenable Cyanide analysis. The blank must be distilled and analyzed with each analytical batch of samples.

9.2.2. CLP:

Each SDG (sample delivery group) must have one method blank processed per matrix and per twenty samples. If the SDG is distilled over multiple "racks", the method blanks must be distilled with the samples. If more than one SDG is on one rack, process one method blank per SDG and per matrix. The solid method blank is called "PBS" (prep blank solid) and the water method blank is called "PBW" (prep blank water).

9.2.2.1. A method blank (PBW/PBS) consisting of 50 mL 0.25N NaOH must be distilled and analyzed with each analytical batch of samples.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. **Non-CLP**:

A midrange LCS consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L secondary source to 50 mL) must be distilled and analyzed with each analytical batch of samples for Total, Amenable, and Free cyanide analysis.

NOTE: A purchased complex cyanide solution may be used instead as the midrange LCS for **Total Cyanide** analysis only.

9.3.2.1. The acceptance limits for drinking water samples is 90 – 110%.

9.3.3. **CLP:**

9.3.3.1. A LCS (which doubles as an ICV) is processed with each batch or each SDG,which ever is more frequent. Even if multiple SDG's are on the "rack", only one LCS (ICV) needs to be distilled for up to a maximum of 20 samples.

NOTE: A "rack" is one setup of cyanide samples and QC, and can encompass 1 – 3 of the midi-distillation units.

9.3.3.2. For CLP waters: the LCS is 4.0 mL of 1.0 ppm secondary source in 50 mL (TV = 80ug/L).

9.3.3.3. For CLP solids: a solid LCS must be processed per SDG or per batch, which ever is more frequent. If more than one batch is processed on a "rack", process a solid LCS for each one. If a SDG has solid samples distilled on a different "rack", then additional solid LCSs will need to be distilled. The value for the LCS changes as the solid lot numbers change each time a new one is ordered. The value is noted on the product certification.

9.3.3.4. If more than one SDG is distilled on one rack, they may be analyzed together (however, it is **preferred** that each SDG be **analyzed separately**). It is also **preferred** that each SDG have its own method blank(s), its own ICV (LCS), its own LCS-S (if samples are solids), and its own sample duplicate and matrix spike. If the SDG has been split over multiple distillation "racks", then you may not have a sample duplicate and matrix spike for each rack. However, an ICV (LCS) is distilled with each "rack". If more than one SDG is distilled on a "rack", then the ICV (LCS) distilled with that rack must be analyzed with each SDG.

9.3.4. Corrective Action for Laboratory Control Samples

9.3.4.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.4.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.3.1. **Non-CLP**

One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.1.1. A MS/MSD consisting of 50 mL or 1.0 g of sample brought up to 50 mL with reagent water and 2.0 mL of the 1.0 mg/L standard (either source) must be distilled with each analytical batch of samples.

9.4.1.2. The acceptance limits for drinking water samples is 90 – 110%.

9.4.2. **CLP:**

Each SDG must have a sample duplicate and a matrix spike processed per matrix. Sometimes this requirement is waived by the client or PM but, if uncertain, make sure that these are done. Usually , the sample to be spiked is assigned, but not always. If a MS/MSD is created in receiving, either batch the MSD and "N/A" the result or have the MSD deleted. In either case, create a sample duplicate for the sample that is spiked when batching. If processing a SDG over more than one "rack", then there is no need to process additional matrix spikes and sample duplicates (unless specifically requested). Typically, only one sample duplicate and matrix spike is required per SDG.

9.4.2.1. Matrix spike for CLP consists of 50 mL or 1.0 g of sample and 100 µg/L spike (5.0 mL of 1.0 mg/L secondary standard).

NOTE: see corrective action section 9.4.3.4 for A-spike

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as MSB and NC for the exception code. The A-spike is not needed in this case.

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.4.3.4. A-spike corrective action: if spike recovery fails criteria, then spike the sample at two times the reporting limit or 2 times the sample concentration, which ever is greater. Examples are shown below.

*A-spike = 2 mL of sample + 2mL of A-spike solution

A-spike solution = 0.8 mL .25N NaOH, +3.2 mL 0.05 ppm 1° standard.

9.5 Control Limits

9.5.1 Control limits are established by the laboratory as described in SOP, NC-QA-0018.

9.5.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest is version easily accessible via the LIMs (QC Browser program).

9.6 Method Detection Limits (MDLs) and MDL Checks

9.6.1 MDLs and MDL Checks are established by the laboratory as described in SOP, NC-QA-0021 and S-Q-003.

9.6.2 MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. **Non-CLP**:

A Low and High standard are distilled from the same source as the calibration curve each day. Prepare the Low standard (0.025mg/L) by diluting 0.125 mL of 10 ppm standard with reagent water to a final volume of 50 mL. Prepare the High standard (0.075 mg/L) by diluting 0.375 mL of 10 ppm standard with reagent water to a final volume of 50 mL. The distilled standards are evaluated against all applicable batch QC.

10.2. **CLP**:

The Low and High standards do not have to be evaluated for CLP.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described

11.3 Summary

11.3.1 Non-CLP

For all non-CLP methods, the sample is distilled/refluxed under acidic conditions for one hour. The released HCN is trapped in 50 mL of 0.25 N NaOH solution.

11.3.2 For CLP and Drinking Water (EPA 335.4) methods, the samples are distilled for one and a half hours. The released HCN is trapped in 0.25 N NaOH solution.

11.4. Sample Preparation Procedure applicable for use with the Midi distillation unit.

11.4.1. Checking for Interferences (For non-CLP samples)

11.4.1.1. Using pH paper strips, check the pH of the sample and record it as >12 or <12 on the analytical logsheet. If pH is <12, the deviation **must be addressed in the project narrative.**

11.4.1.2.For **ALL samples** (non-CLP and CLP samples): Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material.

11.4.1.3. **All Samples**: Test each sample for the presence of chlorine using potassium iodide test strips. Document this on the analytical logsheet.

11.4.2. Complex Cyanide Preparation Procedure [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]: Add 100 mL of water sample or 2.0 g of solid sample and 100 mL reagent water to a plastic, sealable container. Add 10 g NaOH pellets. Mix or tumble for 12-16 hours. Allow solids to settle out. Transfer 50 mL of the solution to the distillation tube. Distill sample (See section 11.4.4.1).

Note: If the sample requires an MS/MSD or MS/Dup, double or triple the volumes as stated above. A blank and LCS should be prepared and analyzed with samples using 10 g NaOH and 100 mL reagent water.

11.4.3. Amenable Cyanide (Chlorinated Aliquot) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.4.3.1.Place 50 mL (waters) or 1.0 g (solids/liquids) into a beaker. Add 50 mL of reagent water to non-waters. Place the beaker on a stir plate and begin mixing. Test the pH of the solution, if less than 12 add 1.25 N NaOH, drop by drop until pH $\geq$ 12. Drop by drop, add calcium hypochlorite until an excess of chlorine is reached. Test for chlorine excess using KI paper. Allow the sample to chlorinate for one hour. At the end of the chlorination period, add about 0.1 to 0.5 g of ascorbic acid to destroy excess chlorine. Test using KI paper. Keep the addition of ascorbic acid to a minimum. Pour the sample into a distillation flask and follow the total cyanide preparation method (Section 11.4.4). Also set up an unchlorinated aliquot of sample (50 mL or 1.0 g) following the total cyanide method.

11.4.4. Total Cyanide

11.4.4.1. Add 50 mL or 1.0 g of the sample and 2 - 3 boiling chips to the distillation tube. Bring the final volume to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH solution to the absorber tube and assemble the scrubber. Assemble the cyanide distillation apparatus.

NOTE: If solid samples are being prepared under the Michigan program, the initial solid weight is 2.5 g

11.4.4.2.Turn on the cooling water to "60". Turn on the vacuum source and adjust the flow such that even stream of air bubbles are in the scrubber tube approximately $^1/_4$ " of foam or 1-2 bubbles per second. At this time add any spiking solutions to the LCS or MS/MSDs samples directly into the inlet tube.

11.4.4.3.In the order stated, add 2.0 mL sulfamic acid solution (not for CLP ILM03.0 samples), 2.5 mL of concentrated sulfuric acid and 2.0 mL of magnesium chloride solution to the distillation tube. Be sure to rinse the inlet tube sparingly with reagent water between and after reagent additions.

Note: Complex cyanide samples require an additional 2.5 mL of sulfuric acid. Be sure the pH is < 2.

11.4.4.4.Flip the heater switch to "on" and turn the dial to an appropriate setting (according to manufacturer's instructions) to allow the apparatus to warm up. Be sure to adjust the air flow and water as necessary. Heat for one hour. After the heating period, the heater will turn off automatically. Allow to cool for fifteen minutes. Keep the vacuum and cooling water on.

11.4.4.5.Disconnect the absorber. Pour solution into a 120 mL snap seal container. Do not rinse the scrubber tube or dilute NaOH in the snap seal. Be sure to properly label the bottles "total" or "amenable" along with sample ID, position and date.

11.4.5. Free Cyanide (Weak and Dissociable) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.4.5.1.Add 50 mL or 1.0 g of sample to the distillation tube. Bring all volumes up to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH to the absorber tube and assemble.

11.4.5.2. Turn on cooling water to "60". Turn on the vacuum source. Add any spiking standards to the appropriate LCS or MS/MSDs at this time. Through the inlet tube, add 1.0 mL of sodium acetate buffer, 1.0 mL of zinc acetate, and 0.25 mL of methyl red indicator. Rinse the inlet tube sparingly with reagent water between and after reagent additions. If the sample is not red, carefully add 1:9 acetic acid dropwise until the sample

does turn red. Check the sample color periodically throughout the distillation hour to ensure the sample stays red.

Reminder: Do not use the complex purchased cyanide solution standard as the LCS for Free cyanide analysis.

11.4.5.3.Flip on the heater switch and turn the dial to "10.5" (This allows the apparatus to warm up). Adjust the air flow and water as needed. Heat for 1 hour. After the heating period, the heater will shut off automatically. Allow the sample to cool for fifteen minutes with the air and water on.

11.4.5.4. Pour the scrubber contents into a 120 mL snap seal bottle. Do not rinse the scrubber. Label the bottle well and be sure to denote that it is a "free" sample distillate.

11.4.6. CLP Cyanide distillation

11.4.6.1. Add 50 mL or 1.0 g of sample to the distillation tube. Bring all volumes up to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH to the absorber tube and assemble.

11.4.6.2. Turn on cooling water to "60". Turn on the vacuum source and adjust the flow such that even stream of air bubbles are in the scrubber tube approximately ¼ " of foam or 1 –2 bubbles per second. At this time add any spiking solutions to the LCS or MS samples directly into the inlet tube.

11.4.6.3. In the order stated, add 2.5 mL of concentrated sulfuric acid and 2.0 mL of magnesium chloride solution to the distillation tube. Be sure to rinse the inlet tube sparingly with reagent water between and after reagent additions.

11.4.6.4. Flip the heater switch to "on" and turn the dial to an appropriate setting to allow the apparatus to warm up. Be sure to adjust the air flow and water as necessary. Heat for one and a half hours. After the heating period, allow to cool for fifteen minutes. Keep the vacuum and cooling water on.

11.4.6.5. Disconnect the absorber. Pour solution into a snap seal container. Do not rinse the scrubber tube or dilute NaOH in the snap seal. Be sure the properly label the snap seals with the sample ID.

11.4.7 Cyanide Unit Clean-up

11.4.7.1 Cyanide distillation unit glassware is very fragile and expensive. It must be handled with care at all times.

11.4.7.2 Disassemble each set-up and rinse the sample down the drain with large amounts of water. Be sure to collect the solids in a screen and dispose of properly.

11.4.7.3 Wash each set-up with soap and hot water. Rinse several times with reagent water. **After preparing samples found to be extremely high in cyanide, a subsequent rinse with 1.0N NaOH, followed by further rinsing with reagent water can help rinse away any residual cyanide. If contamination is suspected, distill 1.0N NaOH through the system.

11.4.7.4 Re-assemble the set-up.

11.4.7.5 Be sure to wash each set up as a separate unit and replace in the same position.

11.4.7.6 If a sample of known high cyanide concentration was distilled in a certain position, be sure to change the appropriate tubing on that position.

11.5 Analytical Documentation

11.5.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2 All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4 Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files

14. POLLUTION PREVENTION

14.1. Using a Midi Cyanide distillation unit saves time and requires less sample and reagents for use.

15. WASTE MANAGEMENT

15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2 Waste Streams Produced by the Method

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

15.2.1.1 Acidic waste. This waste is disposed of in the designated container labeled "Acid Waste".

15.2.1.2 Caustic waste containing Pyridine. This waste is disposed of in a designated container identified as "Pyridine Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References.

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total and Amenable Cyanide, Automated UV; Method 9012A.

16.1.2. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.1, 335.4, and 335.2

16.1.3. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Weak and Dissociable Cyanide; Method 4500-CN-I

16.1.4. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Complex Cyanide; Method 4500-CN-E

16.1.5. USEPA CLP SOW ILM03.0 Section D-Cyanide Midi Distillation

16.1.6. Corporate Quality Management Plan (QMP), current version

16.1.7. STL Laboratory Quality Manual (LQM), current version

16.1.8 STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2 Glassware Washing, NC-QA-0014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-0021 and S-Q-003

16.2.5 Navy/Army SOP, NC-QA-0016

16.2.6 Cyanide, Automated Method NC-WC-0031

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. BASF RFI Requirements

17.1.1. Amendments to this SOP to satisfy BASF RCRA Facility Investigation (RFI) requirements are based on comments from US EPA upon review of site requirements and laboratory procedures.

17.1.2. Amendment to the preparation of **all** samples

17.1.2.1. **Section 7.2.1**: Change 2.51 g of potassium cyanide to 2.11g potassium ferricyanide.

17.1.2.2. **Section 9.4**: The matrix spike/duplicate frequency has been increased from one per batch to one per 7 samples. Consult with the project manger for specific samples to be spiked. The goal is to spike each sampling area at the site at least once.

17.1.3. Amendment to the preparation of **Prussian blue area** samples

17.1.3.1. **Section 7.1.22**: Sodium thiocyanate (reagent grade): Prepare a spiking solution at a concentration 100X greater than the measured cyanide for the appropriate Prussian blue area samples.

17.1.3.2. **Section 9.5**: Three Prussian blue area samples will be spiked with 1 mL of the sodium thiocyanate solution (Section 17.1.3.1) in order to assess the potential impact of thiocyanate in the original samples on the measured cyanide concentration. These matrix spikes do not replace the cyanide matrix spikes mentioned above (Section 17.1.2.2). The increase (if any) in measured cyanide concentration relative to the original sample shall be reported as "percentage increase" using the following equation:

$$\%CN_{increase} = \frac{Conc_{SCN\_spike} - Conc_{unspiked}}{Conc_{unspiked}} \times 100$$

where

$Conc_{SCN\_spike}$ = *cyanide concentration measured in the thiocyanate spiked sample*

$Conc_{unspiked}$ = *cyanide concentration measured in the unspiked sample*

Note: Section 11.3.2.1 of NC-WC-0031 addresses sulfide testing and precipitation.

17.2. Method Deviation (9012A, 335.1, 335.2)

17.2.1. The reflux distillation apparatus used is the midi distillation.

17.2.2. The volume of sample used is reduced to 50 mL vs. 500 mL using the midi-distillation apparatus.

17.2.3. Method of Standard Addition is not performed for samples with matrix interference (sulfides)

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. NC-WC-0031, Rev. 8
Effective Date: 11/08/04
Cover Page

Title: Cyanide, Automated Pyridine-Barbituric Acid
[Methods: SW9012A, EPA335.1, 335.2, SM4500CN-I CLP]

Approvals (Signature/Date):

Technology Specialist	Date 12/14/07	Health & Safety Coordinator	Date 12-14-07
Quality Assurance Manager	Date 12/13/07	Laboratory Director	Date 12/14/07

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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: CYANIDE AUTOMATED, PYRIDINE-BARBITURIC ACID METHOD

(SUPERSEDES: REVISION 7 DATED 05/31/01)

Prepared by:		11/16/04 Date
Reviewed by:	Technology Specialist	11/16/04 Date
Approved by:	Quality Assurance Manager	11/19/04 Date
Approved by:	Environmental Health and Safety Coordinator	11-15-04 Date
Approved by:	Laboratory Director	11/22/04 Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Free Inorganic Cyanide in solids, liquids, and waters. It is based on CLP ILM03.0, CLP ILM04.0, SW846 Method 9012A, 335.2, 335.4 and Standard Method 4500-CN-E. The working linear range is 0.005 - 0.2 mg/L for waters and 0.25 to 10 mg/kg for solids.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

1.3. The associated method codes are CLP ILM03.0 (DV), CLP ILM04.0 (OU), SW846 Method 9012A (QP), EPA 335.1(CF), 335.2 (CG), EPA 335.4 (O2), and Standard Method 4500-CN-E (A4).

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

2.2. The sodium hydroxide solution is analyzed colormetrically on an autoanalyzer using the pyridine-barbituric acid method.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Sulfides interfere, but can be eliminated by treating the sample with cadmium carbonate prior to analysis.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.3. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.4. The acidification of samples prior to extraction/preparation can result in the release of a highly toxic gas, hydrogen cyanide.

5.5. If samples are identified with cyanide concentrations equal to or greater than 200 mg/L, immediately notify the department manager and personnel responsible for hazardous waste shipping. Those samples must be identified as extremely hazardous for other chemists and must receive special attention during disposal.

5.6. Potassium cyanide and sodium cyanide will give off Hyrdogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.7. Cyanide and cyanide salts are extremely toxic. Addition of acid can generate Hydrogen Cyanide gas, which can be extremely dangerous.

5.8. 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

Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Bismuth Nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3 2 Mg/M3-Ceiling	This material will cause burns if comes into contact with the skin or eyes. 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.
Potassium Phosphate	Flammable	None	Inhalation causes severe irritation to the respiratory tract. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. .

Sodium Phosphate		None	Inhalation may cause respiratory tract irritation. Can produce delayed pulmonary edema. Causes mild skin and eye irritation. Injestion may cause gastrointestinal irritation.
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.
Cadmimum Carbonate	Probable carcinogen	0.01 mg/ m^3 as Cd	Ingestion causes increated salivation, choking, vomiting, stomach pains and diahrrea. Inhalation may cause respiratory irritation, nausea and dyspnea.
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.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m^3 – Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, swelling. Greater exposures cause severe burns with possible blindness.

Sodium Cyanide	Poison Corrosive	5 mg/m^3 TWA as CN (skin)	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Chloramine T Hydrate	Poison		May be harmful by inhalation, ingestion, or skin absorption. This material is irritating to mucous membranes and upper respiratory tract. Avoid contact and inhalation.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
Silver Nitrate	Corrosive Poison Oxidizer	0.01 mg/m 3 (TWA) for silver metal dust and fume as 0.02 Ag	This is a corrosive, poisonous material. It will cause burns to any area of contact and is harmful if inhaled. Ingestion may cause death. Contact with other material may cause fire. Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.

1 – Always add acid to water to prevent violent reactions.

2 – Exposure limit refers to the OSHA regulatory exposure limit.

5.9. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned.

5.10. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.11. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents {as well as glassware cleaning procedures that involved solvents such as methylene chloride} should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.12. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.13. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to the EH&S Coordinator and to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Traacs 800 autoanalyzer or discrete autoanalyzer (Konelab)

6.2. Probe 0.016"

6.3. Flowcell 10nm

6.4. Wavelength cell 570nm

6.5. Manifold-Multitest cartridge with cyanide tubing

6.6. 4mL Cuvettes

6.7. 100 mL, 250 mL, 1000 mL volumetric flasks

6.8. Volumetric pipettes: various

6.9. Top loading balance: capable of accurately weighing ± 0.01g.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Cadmium carbonate: powder

7.1.2. 1.25 N sodium hydroxide: Add 50 g of sodium hydroxide pellets (NaOH) to a 1 liter volumetric flask and dilute to volume with reagent water.

7.1.3. Phosphate buffer: Add 136 g of potassium phosphate - monobasic (KH_2PO_4) and 2.8 g of sodium phosphate – dibasic anhydrous (Na_2HPO_4) to 800 mL of reagent water in a 1 liter volumetric flask. Mix, bring to volume with reagent water. Add 3-5 drops of Brij-35 to 100 mL of Buffer prior to using.

7.1.4. Chloramine-T reagent: Add 1.0 g of chloramine-T to a 250 mL volumetric flask and dilute to volume with reagent water. Prepare fresh daily.

7.1.5. Pyridine reagent: Add 15.0 g of barbituric acid to a 1 liter volumetric flask. Add 75 mL of pyridine and 15 mL of concentrated hydrochloric acid (HCl) and mix. Bring to volume with reagent water and store at 4°C ± 2°C in an amber glass bottle.

Note: the pyridine barbituric acid may be purchased commercially. Filter 50 ml of the pyridine barbituric acid and bring up to 250 ml with DI water.

7.1.6. 0.25 N sodium hydroxide: Add 200 mL of 1N NaOH to a 1 liter volumetric flask and dilute to volume with reagent water.

Note: 0.25 N NaOH may be purchased instead.

7.1.7. Brij-35

7.1.8. 30% Brij solution: Add 2 mL Brij-35 to 1000 mL reagent water.

7.1.9. Rhodanine indicator, purchased.

7.1.10. 0.0192 N silver nitrate, purchased

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months.

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow 7.2.1 using an alternate source of Potassium Cyanide (KCN).

Note: This stock standard may also be purchased.

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1. If using the 1.25 N NaOH: pipet the appropriate amount of cyanide standard into 100 mL volumetric and add 20 mL 1.25N NaOH to each calibration standard (except the 1.0 mg/L) and bring to volume with reagent water.

If using the purchased 0.25N NaOH: pipet the appropriate amount of cyanide standard into 100 mL volumetric and bring to volume with 0.25N NaOH.

Prepare weekly.

Concentration CN-	mL CN-	Final Volume
10 mg/L	1 mL of 1000 mg/L	100 mL
1.0 mg/L (secondary only)	10 mL of 10 mg/L	100 mL
*0.2 mg/L	2 mL of 10 mg/L	100 mL
*0.1 mg/L	1 mL of 10 mg/L	100 mL
*0.05 mg/L	0.5 mL of 10 mg/L	100 mL

*0.025 mg/L	0.25 mL of 10 mg/L	100 mL
*0.01 mg/L	0.1 mL of 10 mg/L	100 mL
*0.005 mg/L	0.05 mL of 10 mg/L	100 mL

*Denotes calibration standards

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Solid samples are not chemically preserved. Water samples are preserved with NaOH to a pH > 12. All samples are stored at 4°C ± 2°C in plastic or glass containers.

8.2. The holding time for **non-CLP** samples is fourteen days from sampling to analysis.

8.3. The holding time for **CLP** samples is twelve days from receipt to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. **Non –CLP**:

9.1.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.1.2. **CLP**

9.1.2.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS/ICV, Method Blank, Sample Duplicate and Matrix Spike) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.1.2.2. A Chain of Custody (COC) is needed for each SDG.

9.2. Method Blank

9.2.1. Non-CLP

9.2.1.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water or 0.25N NaOH containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.1.2. A method blank consists of 50 mL 0.25N NaOH for Total Cyanide analysis or 50 mL reagent water for Free and Amenable Cyanide analysis and must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-0032 for distillation instructions.

9.2.2. CLP

9.2.2.1. Each SDG (sample delivery group) must have one method blank (MB) processed per matrix and per twenty samples. If the SDG is distilled over multiple "racks", the method blanks must be distilled with the samples. If more than one SDG is on one rack, process one method blank per SDG and per matrix. The solid method blank is called "PBS" (prep blank solid) and the water method blank is called "PBW" (prep blank water).

NOTE: A "rack" is one setup of cyanide samples and QC, and can encompass 1-3 of the midi-distillation units.

9.2.2.2. A method blank (PBW/PS) consisting of 50 mL 0.25N NaOH must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-0032 for distillation instructions.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other

considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS from an independent source must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. Non-CLP

9.3.2.1. A midrange LCS consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L to 50 mL) must be distilled and analyzed with each analytical batch of samples for Total, Amenable, and Free cyanide analysis. See SOP NC-WC-0032 for distillation instruction.

NOTE: A purchased complex cyanide solution may be used instead as the midrange LCS for **Total Cyanide** analysis only.

9.3.2.2. The acceptance limits for drinking water samples, Method 335.4, is 90 – 110%.

9.3.3. CLP

9.3.3.1. A LCS (which doubles as an ICV) is processed with each batch or each SDG, which ever is more frequent. Even if multiple SDG's are on the "rack", only one LCS (ICV) needs to be distilled for up to a maximum of 20 samples.

NOTE: A "rack" is one setup of cyanide samples and QC, and can encompass 1 – 3 of the midi-distillation units.

9.3.3.2. For CLP waters: the LCS is 4.0 mL of 1.0 ppm secondary source in 50 mL (TV = 80ug/L).

9.3.3.3. For CLP solids, a solid LCS must be processed per SDG or per batch, which ever is more frequent. Weigh out 0.05g of the solid LCS sample. If more than one SDG is processed on a "rack", process a solid LCS for each one. If a SDG has solid sample distilled on a different "rack", then additional solid LCS's will need to be distilled. The value for this LCS changes as the solid lot number changes each time a new one is ordered. The value is noted on the product certification.

9.3.3.4. If more than one SDG is distilled on one rack, they may be analyzed together (however, each SDG **must** be **analyzed separately with a separate PBW**). It is also **preferred** that each SDG have its own method blank(s), its own ICV (LCS), its own LCS-S (if samples are solids), and its own sample duplicate and matrix spike. If the SDG has been split over multiple distillation "racks", then you may not have a sample duplicate and matrix spike for each rack. However, an ICV (LCS) is distilled with each "rack". If more than one SDG is distilled on a "rack", then the ICV (LCS) distilled with that rack must be analyzed with each SDG.

9.3.4. Corrective Action for LCS

9.3.4.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.4.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. Non-CLP

9.4.1.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix

on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.1.2. A MS/MSD consisting of 50 mL or 1.0 g sample and 0.04 mg/L spike (2.0 mL of 1.0 mg/L to 50 mL) will be distilled and analyzed with every batch. See SOP NC-WC-0032 for distillation instructions. This is also the same value for CLP.

9.4.1.3. The acceptance limits for drinking water samples, Method 335.4, is 90 – 110%.

9.4.2. **CLP**

9.4.2.1. Each SDG must have a sample duplicate and a matrix spike processed per matrix. Sometimes this requirement is waived by the client or PM but, if uncertain, make sure that these are done. Usually , the sample to be spiked is assigned, but not always. If a MS/MSD is created in receiving, either batch the MSD and "N/A" the result or have the MSD deleted. In either case, create a sample duplicate for the sample that is spiked when batching. If processing a SDG over more than one "rack", then there is no need to process additional matrix spikes and sample duplicates (unless specifically requested). Typically, only one sample duplicate and matrix spike is required per SDG.

9.4.2.2. Matrix spike for CLP consists of 50 mL or 1.0 g of sample and 100 µg/L spike (5.0 mL of 1.0 mg/L secondary standard).

NOTE: see corrective action section 9.4.3.4 for A-spike

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as "amount" MSB. The Exception Code is changed to NC. The A-spike is not needed in this case.

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.4.3.4. A-spike corrective action: if matrix spike recovery fails criteria, then spike the sample at two times the reporting limit or 2 times the sample concentration, which ever is greater. Examples are shown below.

*A-spike = 2 mL of sample + 2mL of A-spike solution

A-spike solution = 0.8 mL .25N NaOH, +3.2 mL 0.05 ppm 1° standard

9.5. QC Acceptance Criteria

9.5.1. Control limits are established by the laboratory as described in NC-QA-0018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest is version easily accessible via the LIMs (QC Browser program.).

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOPs S-Q-003 and NC-QA-0021.

9.6.2. MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. The instrument is calibrated using six cyanide standards and a blank at the time of analysis. Non- CLP samples are processed in mg/L and CLP samples are in ug/L.

10.2. Initial Calibration

10.2.1. Non-CLP

10.2.1.1. The instrument is calibrated at the beginning of each run and is verified at the beginning of the run by using a midrange ICV. The ICV is composed of the 0.1 ppm secondary standard. The ICV must not vary from the original curve by more than ± 15% or recalibration is required. For drinking water samples, Method 335.4, the criteria is ± 10%. The correlation coefficient of the original curve must be ≥ 0.995 or recalibration is required.

10.2.2. CLP

10.2.2.1. The instrument is calibrated at the beginning of each run and is verified at the beginning of the run by analyzing the ICV/LCS. The ICV is composed of 4.0 mL of 1.0 ppm secondary standard (TV = 80 ug/L). The correlation coefficient of the original curve must be ≥ 0.995 or recalibration is required.

10.3. Continuing Calibration

10.3.1. The run is checked every ten samples and at the end of the run using a midrange CCV to verify continued linearity. It cannot vary from the original curve by more than ± 15% or recalibration is required. For drinking water samples, Method 335.4, the criteria is ± 10%. The previous 10 samples must be reanalyzed and bracketed by a CCV that passes criteria. The CCV is composed of the 0.1 mg/L primary standard for non-CLP runs and the 100 ug/L for CLP runs.

10.3.2. System cleanliness is checked every ten samples and at the end of the run using a CCB. It cannot contain the analyte of interest above the reporting limit or recalibration is required. The previous 10 samples must be reanalyzed and bracketed by a CCB that passes criteria. The CCB is 0.25N NaOH.

NOTE: Base and gain values may change with new reagents, standards, tubing changes, or board cleaning. These values are for reference only.

10.4. High and Low Standard

10.4.1. The distillation technique is checked by distilling a high and low standard and comparing the values obtained to the standard curve. The method recommends that the HI/LO standards be compared to the curve with a +/-10% agreement. The HI/LO standards are evaluated against all applicable batch QC.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. See cyanide distillation SOP: NC-WC-0032.

11.3.1.1. Non-CLP

11.3.1.1.1. The sample is distilled/refluxed under acidic conditions for one hour. The released HCN is trapped in 50 mL of 0.25 N NaOH solution. EPA Method 335.4 says to reflux for one and a half hours.

11.3.1.2. CLP

11.3.1.2.1. The sample is distilled/refluxed under acidic conditions for one and a half hours. The released HCN is trapped in 50 mL of 0.25 N NaOH solution.

11.3.2. Sample Preparation Procedure

All Samples:Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of

cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material.

NOTE: Water should be tested prior to distillation

11.4. Sample Analysis

11.4.1. Summary

11.4.1.1. The sample distillates are analyzed on the autoanalyzer for cyanide using the automated pyridine-barbituric method.

11.4.2. Recommended Instrument Conditions

11.4.2.1. See Manufacturer's information for operation instructions.

11.4.2.2. Start Up Solutions (and Wash Solutions)

11.4.2.2.1. All lines in 0.25N NaOH solution:

11.4.2.2.2. Then all lines in 30% Brij solution.

11.4.2.3. Running Solutions

11.4.2.3.1. Two DI water lines (orange/green) in 30% Brij solution

11.4.2.3.2. Red/Red in pyridine reagent

11.4.2.3.3. Orange/red in chloramine-T reagent

11.4.2.3.4. White/white in phosphate buffer solution

11.4.2.4. Base and Gain

11.4.2.4.1. Performed on the 0.2 mg/L standard

11.4.2.4.2. Approximate values: Base = 75 Gain = 150

*These values are for reference only and may change with new reagent or standard preparations.

11.4.2.5. Trouble shooting for poor Base and Gain or baseline noise

11.4.2.5.1. Place reagent lines 1-3 back in Brig solution and run on high for five minutes. Place all 5 lines (1-5) and probe line in 1.25 N NaOH solution and run on high for five minutes. Place lines back in appropriate "start-up" solutions and run on high for five minutes or make fresh new reagents.

Place lines back in reagents and perform another base and gain when the baseline has stabilized.

11.4.3. Sample Analysis Procedure

11.4.3.1. See Manufacturer's information for operating instructions.

11.4.3.2. A calibration curve is analyzed at the beginning of each run. The correlation coefficient must be > 0.995 to continue.

11.4.3.3. The ICV (from the secondary source) and the ICB is analyzed at the beginning of every run. CCV's (from the primary source) and CCB's are analyzed at the end and between every 10 samples.

11.4.3.4. Sample distillates higher than the highest calibration standard (0.2 mg/L) must be diluted with 0.25 N NaOH and re-analyzed.

11.4.3.5. Any samples analyzed after a high sample must be re-analyzed if carryover is suspected.

11.5. Tray Protocol (Run Order)

Normal Tray Protocol

1. Calibration
2. MRL
3. ICV (secondary source)
4. ICB (CCB)

5. Prep Blank
6. LCS
7. LOW
8. HI
9. UP to 6 more samples
10. MRL
11. CCV
12. CCB
13. Up to 10 samples
14. MRL
15. CCV
16. CCB

CLP Tray Protocol-Water

1. Calibration
2. Distilled ICV/LCS
3. ICB
4. CCV
5. CCB
6. PBW
7. Sample

8. Sample duplicate
9. Sample MS
10. Up to 6 more samples
11. CCV
12. CCB
13. Up to ten more samples
14. CCV
15. CCB

CLP Tray Protocol-Solid

1. Calibration standards
2. Distilled ICV
3. ICB
4. CCV
5. CCB
6. PBS
7. LCSS
8. Sample
9. Sample duplicate
10. Matrix Spike
11. Up to 5 more samples

12. CCV

13. CCB

14. Up to ten more samples

15. CCV

16. CCB

11.6. Analytical Documentation

11.6.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.6.2. All standards and reagents are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.6.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.6.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. $$\textit{Total Cyanide, mg / L} = \frac{\textit{mg / L } CN^- \textit{ from printout x 50}}{\textit{mL of sample distilled}} \textit{ x D}$$

12.2. $$\textit{Total Cyanide, mg / kg} = \frac{\textit{mg / L } CN^- \textit{ from printout x 50}}{\textit{g of sample distilled.}} \textit{ x D}$$

12.3. $$\textit{Amenable Cyanide, mg / L} = \textit{Total } CN^- \textit{ (mg / L) - Chlorinated } CN^- \textit{ (mg / L)}$$

Where:

mg/L = ***can also be mg/kg***

D = ***Dilution Factor*** = $\frac{\textit{Final Volume of Dilution}}{\textit{Volume of Sample Distillate Used}}$

NOTE: Free cyanide has the same calculations as Total cyanide

12.4. LCS Recovery:

$$\frac{\textit{Instrument Value}}{0.04(\textit{true})} \times 100 = \%\ \textit{Recovery}$$

12.5. CCV Recovery:

$$\frac{\textit{Instrument Value}}{0.025\ (\textit{true})} \times 100 = \%\ \textit{Recovery}$$

NOTE: CCV recovery must be between 85 - 115% for data to be acceptable. If CCV recovery is not within these limits, reanalysis is required 335.4 says 10%.

12.6. ***MS/MSD Recovery for Waters and solids:***

$$\frac{A - B}{0.040\ (\textit{true})} \times 100 = \%\ \textit{Recovery}$$

Where:

A = ***Instrument value MS/MSD***

B = ***Sample instrument value***

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

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

15.2.2. Alkaline material from the auto-analyzer. This waste is collected in the lab in a container labeled "Pyridine Waste".

15.2.3. Acidic waste This waste is collected in the lab in a container labeled "Acid Waste".

15.2.4. Caustic waste containing Pyridine. This waste is collected in the lab in a containers labeled "Pyridine Waste".

15.2.5. Wastewater contaminated with Pyridine. This waste is collected in the lab in a containers labeled "Pyridine Waste".

15.2.6. Wastewater containing 1.5% Pyridine This waste is collected in the lab in a containers labeled "Pyridine Waste".

15.2.7. Aqueous analytical waste, neutral to slightly basic, contaminated with 3% pyridine. This waste is collected in the lab in a container labeled "Pyridine Waste".

15.2.8. Filter paper contaminated with lead sulfideThis waste is placed in a container labeled "Solid Waste".

15.2.9. Aqueous rinsates from distillation tube clean upThis waste is collected in the lab and disposed of in a container labeled "Acid Waste".

15.2.10. Miscellaneous solid waste contaminated with sample residue, acids, caustics and reagents used in this SOP. A weigh tin is the only type of this waste generated. The tin is rinsed with water and disposed of in a container labeled "Solid Waste".

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste Method 9012 A, and its updates

16.1.2. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.2, March 1983

16.1.3. EPA 600; Determination of Total Cyanide by Semi-Automated Colorimetry, 335.4, Revision 1.0, August 1993.

16.1.4. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition: Weak and Dissociable Cyanide; Method 4500-CN-E

16.1.5. USEPA CLP SOW ILM03.0 and ILM04.0, Section D - Cyanide Midi Distillation

16.1.6. Corporate Quality Management Plan (QMP), current version.

16.1.7. STL Laboratory Quality Manual (LQM), current version

16.1.8. STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, S-Q-003 and NC-QA-0021

16.2.5. Navy/Army SOP, NC-QA-0016

16.2.6. NC-WC-0032, Cyanide Preparation Method

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The reporting limit (RL) is 0.01 mg/L for waters (50 mL used) and 0.50 mg/kg for solids (1.0 g used). The reporting limit for solid samples performed only under the Michigan program is 200 µg/kg for solids (2.5 g used). The lowest level of the calibration curve can be used as the reporting limit upon request.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide for poor Base and Gain or baseline noise

17.2.1. Place reagent lines 1-3 back in Brig solution and run on high for five minutes. Place all 5 lines (1-5) and probe line in 1.25 N NaOH solution and run on high for five minutes. Place lines back in appropriate "start-up" solutions and run on high for five minutes.

Place lines back in reagents and perform another base and gain when the baseline has been stabilized.

17.3. Method Deviation (9012A/335.2)

17.3.1. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

Appendix I - Cyanide Standardization

1. Pipet 10.0 mL of the 1000 ppm stock cyanide standard into a 250 mL Erlenmeyer flask and add 90 mL of reagent water.
2. Add 0.5 mL (10 drops) of Rhodanine indicator.
3. Titrate with 0.0192 N silver nitrate (using a micro burette) until the color changes from yellow to pink/orange.
4. Titrate a blank (100 mL reagent water) following steps 2 and 3.
5. Calculation:

$$\textbf{Cyanide, mg / L} = \frac{\textbf{(A - B) (1000)}}{\textbf{mL Cyanide Solution (10)}}$$

Where:

A = mL titrant for standard

B = mL titrant for blank

6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

Title: SULFIDE

[SW846 Method 9030B, EPA 376.1, and SM 4500-S2-E]

Approvals (Signature/Date):

Olguita Colon	10/13/07		10-16-07
Technology Specialist	Date	Health & Safety Coordinator	Date
Dorothy J. Reeson	10/17/07		10/16/07
Quality Assurance Manager	Date	Laboratory Director	Date

This SOP was previously identified as SOP No. NC-WC-0060, Rev 4, dated 09/24/04

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of the concentration of Sulfide in waters, liquids, solids, and sludges. It is based on SW846 Method 9030B, Methods for Chemical Analysis of Water and Wastes EPA 376.1, and SM 4500-S2-E. The working range is 1 to 30 mg/L for waters and 10-650 mg/kg for solids and sludges.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

1.3. The associated QuantIMS method codes are TV (9030B), OG (4500-S2-E), and CT (376.1).

2. SUMMARY OF METHOD

2.1. For acid soluble sulfide samples, separation of sulfide from the sample matrix is accomplished by the addition of sulfuric acid to the sample. The sample is heated to 70^0C and the hydrogen sulfide (H_2S) which is formed, is distilled under acidic conditions and carried by a nitrogen stream into zinc acetate scrubbing bottles where it is precipitated as zinc sulfide.

2.2. For acid-insoluble sulfide samples, separation of sulfide from the sample matrix is accomplished by suspending the sample in concentrated hydrochloric acid by vigorous agitation. Tin (II) chloride is present to prevent oxidation of sulfide to sulfur by the metal ion (as in copper (II)), by the matrix, or by dissolved oxygen in the reagents. The prepared sample is distilled under acidic conditions at 100^0C under a stream of nitrogen. Hydrogen sulfide gas is released from the sample and collected in gas scrubbing bottles containing zinc (II) and a strong acetate buffer. Zinc sulfide precipitates.

2.3. An excess of iodine is added to a sample which oxidizes the Sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Reducing substances such as thiosulfite, sulfites, and various organic compounds cause interferences, but treatment with zinc acetate solution will eliminate some of these interferences. (Use approximately 15 drops of 2 N zinc acetate per 500 mL of sample if not already preserved with it.)

4.3. Samples that contain strong oxidizers or reducers will interfere with this method.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. 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.
Formaldehyde	Poison	260 Mg/M^3 TWA	Inhalation of vapors may cause respiratory irritation leading to frequent bronchial infection. Eye contact causes redness, watering, and itching. Skin contact causes itching, scaling, and reddening or blistering.
Iodine	Poison Corrosive Oxidizer	0.1 ppm-Ceiling	Vapors severely irritate and can burn the mucous membranes and respiratory tract. Liquid contact may cause blistering burns, irritation, and pain. Vapors may be severely irritating to the skin. Vapors are severely irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/ M^3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sodium Hydroxide	Corrosive	2 Mg/ M^3 - 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/ M^3 - TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose, 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.
Sodium Sulfide	Corrosive	10 ppm-TWA 15 ppm-STEL	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
1 – Always add acid to water to prevent violent reactions.			
2- Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. Inhalation of HS gas may be fatal.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.9. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.10. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.11. Sulfide titration for method 376.1 must be performed in a fume hood.

6. EQUIPMENT AND SUPPLIES

6.1. Volumetric pipettes: various

6.2. Buret: 25 mL Class A

6.3. Erlenmeyer flasks: 500 mL

6.4. Graduated cylinder: 250 mL

6.5. Top loading balance: capable of accurately weighing ± 0.01 g

6.6. Volumetric flasks: various

6.7. Vacuum pump, filter and flask

6.8. Whatman #4 filters

6.9. Distillation apparatus containing: 250 mL addition funnel, 500 mL – 3-neck reaction flask, sparging tube and 1 – 500 mL Erlenmeyer Flask.

6.10. Stirring / Hot Plates

6.11. Crystallizing dishes

6.12. Ottowa sand

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. (1:1) Hydrochloric Acid: Add 250 mL concentrated hydrochloric acid (HCl) to 250 mL of reagent water.

7.1.2. Starch Indicator: Add 10 mL of reagent water to 5 g starch (potato) and mix. Add starch mixture to 500 mL of boiling reagent water. Mix, cool, and store in a well-labeled squirt bottle. Alternately, use purchased starch solution.

7.1.3. 0.025 N Sodium Thiosulfate (stored in dessicator): Add 0.4 g NaOH and 6.205 g of sodium thiosulfate ($Na_2S_2O_3 \bullet 5H_2O$) to 500 mL of reagent water in a 1 liter volumetric. Dilute to volume with reagent water. Store in a dark container. Also available commercially.

7.1.3.1. Standardization of 0.025 N Sodium Thiosulfate Solution: *To make 0.025N Biodate Solution, dissolve 0.462 g $KH(IO_3)_2$ in 500 mL with reagent water. Weigh 2 g KI in a 500 mL Erlenmeyer flask. Add 100 to 150 mL reagent water, 5 drops H_2SO_4 and 20 mL biodate solution using a volumetric pipet. Dilute to 200 mL with reagent water. Titrate with Sodium Thiosulfate. When a pale straw yellow color is reached, add 1-2 mL starch. Continue titrating from a blue to a clear end point.

Note: Biodate Solution may be purchased.

Calculation:

$$Na_xS_2O_3\ Normality = \frac{(a)\ (b)}{c}$$

Where:

a = mLs Biodate (20 mL)

b = Normality Biodate (0.025N)

c = mLs of $Na_2S_2O_3$ used to titrate Repeat two more times

7.1.4. 0.0282 N Iodine Solution: Add 20 g KI (potassium iodide) and 3.2 g iodine to a 1 liter volumetric flask. Add 500 - 700 mL of reagent water and dissolve. Dilute to

volume with reagent water. Store in a dark container. Also available commercially.

7.1.4.1. Standardization 0.0282 N Iodine Solution: Perform three method blanks daily. Refer to method blank section in SOP.

Calculation:

$$\textit{Normality Iodine} = \frac{(\textit{Normality } Na_2S_2O_3)(\textit{mL of titrant } Na_2S_2O_3)}{\textit{20 mL Iodine}}$$

7.1.5. 2N Zinc Acetate: Dissolve 220 g of zinc acetate in 870 mL of reagent water and dilute to 1 liter with reagent water.

7.1.6. Formaldehyde (37% solution), CH_20). This solution is commercially available.

7.1.7. Zinc Acetate for the Erlenmeyer flasks:

7.1.7.1. For acid-soluble Zinc: Zinc acetate solution (approximately 0.5M). Dissolve 110g Zinc acetate, dihydrate $NaC_2H_3O_2$, in 800 mL of reagent water. Add 1 mL concentrated hydrochloric acid and dilute to 1 liter.

7.1.7.2. For acid-insoluble sulfides: Zinc acetate/sodium acetate buffer. Dissolve 100 g sodium acetate, $NaC_2H_3O_2$, and 11 g zinc acetate dihydrate in 800 mL of reagent water. Add 1 mL concentrated hydrochloric acid and dilute to 1 liter. The resulting pH should be 6.8

7.1.8. Sulfuric acid – 50%. Place 450 mL of reagent water in a volumetric flash. **Slowly** add 500 mL concentrated Sulfuric Acid (H2SO4). **Use extreme caution; this is an exothermic reaction and will create excess heat**. This solution is commercially available.

7.1.9. Hydrochloric Acid, 9.8N, for **acid-insoluble sulfides**. Place 200 mL of reagent water in a 1-liter beaker. Slowly add concentrated HCl to bring the total volume to 1 liter.

7.1.10. Tin (II) chloride, $SnCl_2$, granular

7.2. Standards

7.2.1. Laboratory Control Sample

7.2.1.1. 2000 ppm Sulfide: Add 3.5 g of sodium sulfide to 100 mL of reagent water in a 250mL volumetric flask. Dilute to volume with reagent water. The sulfide standard must be verified each working day. If the resulting value is <75% of the original standard, a new solution must be prepared.

7.2.2. Matrix Spike Standard

7.2.2.1. Prepare a midrange matrix spike standard as described in 7.2.1 for use as a MS/MSD.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Waters are preserved to a pH > 9 with NaOH and zinc acetate. Non-water samples are unpreserved. All matrices are stored at 4°C ± 2°C in plastic or glass containers.

8.2. The holding time is seven days from sampling to analysis for aqueous samples. Samples must be analyzed immediately if arrived in the laboratory without proper chemical preservation.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. A reagent water blank consisting of 250 mL of reagent water, or 30 g Ottowa sand and 200 mL DI for solids, must be analyzed with each analytical batch of samples.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS is prepared by adding 2.5 mL for Methods 376.1 and 4500-S2-E, and 2.0 mL for Method 9030B (water samples) or 2.0 mL (solid samples) of 2000 ppm sulfide standard to a flask. This standard must be analyzed with each analytical batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. The only exception is that if the LCS recoveries are biased high and the associated sample is ND for the parameter(s) of interest, the batch is acceptable. **This must be addressed in the project narrative.**

9.3.3.3. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD

results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. An MS/MSD consisting of 30 g or 250 mL of the sample and 2.5 mL for Methods 376.1 and 4500-S2-E, or 2.0 mL for Method 9030B of 2000 ppm sulfide standard should be analyzed with every 20 samples.

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as "amount" MSB. The Exception Code is changed to NC. The following two footnotes will appear on the report page 'NC The recovery and/or RPD were not calculated." "MSB The recovery and RPD were not calculated because the sample amount was greater than four times the spike amount."

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the laboratory limits.

9.5. Control Limits

9.5.1. Control limits are established by the laboratory as described in SOP NC-QA-0018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. The latest version is easily accessible via the LIMs (QC Browser program).

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOP NC-QA-0021 and S-Q-003.

9.6.2. MDLs are easily accessible via the LIMs (QC Browser program).

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Not Applicable

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Aqueous Sample Method 9030B

11.3.1.1. For an efficient distillation, the mixture in the distillation flask must be of such a consistency that the motion of the stirring bar is sufficient to keep the solids from settling. The mixture must be free of solid objects that could disrupt the stirring bar. Prepare the sample using one of the procedures in this section then proceed with the distillation step.

11.3.1.2. If the sample is aqueous, shake the sample container to suspend any solids, then quickly decant the appropriate volume (250 mL) of the sample to a graduated cylinder. Transfer the contents of the graduated cylinder into the reaction flask.

11.3.1.3. If the sample is aqueous, but contains a large proportion of solids, the sample may be roughly separated by phase and the amount of each phase measured and weighed to the nearest milligram into the distillation flask in proportion to their abundance in the sample. Reagent water may be added up to a total volume of 250 mL.

11.3.2. Solids and Waste Samples, Method 9030B

11.3.2.1. Weigh out 30 g +/- 0.1 g of homogenized sample and put into the reaction flask.

11.3.2.2. Samples that are not water miscible (oils, various solvents) cannot be analyzed using this method.

11.3.3. 9030B Distillation

11.3.3.1. Acid Soluble Sulfides:

11.3.3.1.1. Add 30g +/- 0.1g of solid sample plus 200 mL of reagent water or 250 mL of aqueous sample and a stir bar to the reaction flask.

11.3.3.1.2. Attach the reaction flask to the distillation apparatus such that the bottom of the reaction flask does not touch the bottom of the crystallizing dish (submerge approximately 1/3 of the flask in the warm water).

11.3.3.1.3. Add 100 mL of 50% H2SO4 (7.1.8) to the addition flask and place in the center neck of the reaction flask. Attach the nitrogen flow line to the top of the addition flask.

11.3.3.1.4. Prepare one (1) gas trap bottles for each distillation setup by adding 200 mL of reagent water, 40 mL of zinc acetate buffer (Section 7.1.7.1) and 10 mL of formaldehyde (Section 7.1.6) to each trap.

11.3.3.1.5. Connect the trap and insert the trap arm into the right neck of the reaction flask. Turn on the nitrogen to approximately 5 psi and adjust the flow to 3 – 5 bubbles per second and purge for 15 minutes.

11.3.3.1.6. Add spike and LCS solution by removing the trap arm and pipetting the spike solution, below the surface of the water. Replace the trap arm and re-establish the flow of nitrogen, purge for 5 minutes.

11.3.3.1.7. Open the addition funnel to add the sulfuric acid (Section 7.1.8) drop by drop. Do not open the stopcock fully. Distill the sample for 90 minutes, maintaining 70^0C in the water bath.

11.3.3.1.8. Fill the crystallizing dish with 650 – 700 mL of reagent water and place on the hotplate/stirrer. Turn on the hotplate to a setting of "6" for approximately 15 – 20 minutes. Turn down to a setting of "4" to maintain a temperature of 70^0C, +/- 5^0C.

11.3.3.2. Acid-Insoluble Sulfide:

11.3.3.2.1. As the concentration of HCl during distillation must be within a narrow range for successful distillation of H2S, the water content must be controlled. It is imperative that the final concentration of HCl in the distillation flask be about 6.5N and that the sample is mostly suspended in the fluid by the action of the stirring bar. This is achieved by adding 50 mL of reagent water, including water in the sample, 100 mL of 9.8N HCl, and the sample to the distillation flask. Solids, which absorb water and swell, will restrict fluid motion and, therefore, lower recovery will be obtained. Such samples should be limited to 25 g dry weight.

11.3.3.2.2. If the matrix is aqueous, then a maximum of 50 g of the sample may be used. No additional water may be added.

11.3.3.2.3. If the matrix is dry solid, use 30 g +/- 0.1 g of the sample and add 50 mL of reagent water.

11.3.3.2.4. Add 5 g SnCl to each distillation flask

11.3.3.2.5. Assemble the distillation apparatus. Place 200 +/- 4.0 mL of zinc acetate/sodium acetate buffer solution and 10.0 mL +/- 0.02 mL of 37% formaldehyde in each Erlenmeyer flask. Add 40 mL DI water.

11.3.3.2.6. Add 100 +/- 1.0 mL of 9.8N HCL to the addition funnel. Connect the nitrogen line to the top of the funnel and turn the nitrogen on to pressurize the dropping funnel headspace.

11.3.3.2.7. Set the nitrogen flow at 25 mL/min. The nitrogen in the Erlenmeyer flask should bubble at about five bubbles per second. Purge the oxygen from the system for about 15 minutes.

11.3.3.2.8. Turn on the magnetic stirrer. Set the stirring bar to spin as fast as possible. The fluid should form a vortex. If not, the distillation will exhibit poor recovery. Add all the HCl from the dropping funnel to the flask.

11.3.3.2.9. Heat the water bath to the boiling point (100^0C). the sample may or may not be boiling. Allow the purged distillation to proceed for 90 minutes at 100^0C. **Note**: **Watch water dishes so they don't go dry!**

11.4. Sample Analysis

11.4.1. Sample Analysis Procedure

11.4.1.1. Methods 376.1 and 4500-S2-E

11.4.1.1.1. Place 250mL of homogenized sample in a 500 mL Erlenmeyer flask. At this time, add any spiking solutions if necessary for the filtered water samples.

11.4.1.1.2. Add 20.0 mL .028 N Iodine solution and 1-2 mL 1:1 HCl solution (watch for fumes). Check the pH prior to titration to make sure it is less than 2. If the pH is not <2, add additional acid. Add 250 mL reagent water only for the water samples and mix.

11.4.1.1.3. Add 1 squirt (1-2 mL) of starch indicator and mix. Titrate from blue to clear with .025 N sodium thiosulfate titrant. Record the amount of titrant used on the analytical logsheet.

Note: Some matrices may be turbid or colored and the color change from blue to clear may not be easily seen. In this case, look for a shade change.

11.4.1.2. 9030B/9034 Titrations

11.4.1.2.1. Titrate the scrubber solution in the Erlenmeyer.

11.4.1.2.2. Add 20 mL of 0.028 N iodine solution under the liquid level in the Erlenmeyer, and 1-2 mL of 1:1 HCl solution. (More HCl is needed for insoluble 9030B.) Check the pH prior to titration to make sure it is less than 2.

11.4.1.2.3. Add 1 squirt of starch indicator and mix. Titrate from blue to clear with .025 N sodium thiosulfate titrant.

Note: Because of the buffer and the formaldehyde, the titration will take between 15 – 30 minutes. Add titrant slowly to allow the reaction to take place. Overtitrating could be an issue if the titrant is added too quickly.

11.4.1.2.4. After adding 20 mL iodine, the color should be orange/red. If the color remains yellow, add additional 10 mL aliquots until the orange/red color persists (adjust the calculation

accordingly). If the sample requires more than 60 mL of iodine, the sample must be re-prepped at a smaller dilution. The iodine should turn a yellow-orange color when added to the sample after the addition of the reagent water. If it does not, the sample may be high in sulfide and less sample should be used, or more iodine for 9030B.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, which may be in electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calculations

$$\textit{Sulfide, mg / L or mg / kg} = \frac{[(A \times B) - (C \times D)] \times 16{,}000}{\textit{mL or g of sample used}}$$

Where:

A = mL of iodine solution

B = Normality of iodine solution

C = mL of sodium thiosulfate titrant

D = Normality of sodium thiosulfate titrant

$$\textit{Sulfide, mg / L or mg / kg} = \frac{(20 - \textit{mL titrant}) \times 400}{\textit{mL or g of sample used}}$$

12.1.1.

$$LCS\,\%\ \text{Recovery} = \frac{\text{mg/L (from 12.1.1)}}{\text{20 (true)}} \times 100$$

Note: The true value of the standard is determined daily.

12.1.2.

$$\text{MS/MSD}\ \%\ \text{Recovery} = \frac{\text{A - B}}{\text{20 (waters) or 1000 (solids)}} \times 100$$

Where:

A = (20 - mL titrant for MS/MSD) x 400

B = Concentration from 12.1.1 x mL or g of sample used

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica North Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.3. Waste Streams Produced by the Method

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

15.3.1.1. Acidic waste generated by sample titration. Waste material is disposed of in acid waste containers located in the lab.

15.3.1.2. Aqueous acidic material from the distillation double- necked flask is collected in 5-gallon containers and taken to the neutralization area for neutralization.

15.3.1.3. Aqueous sample waste containing formaldehyde, sodium thiosulfate, and iodate. Waste is disposed of in waste containers specific to this test method located in the laboratory.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods of Evaluating Solid Waste, Third Edition, Sulfide, Method 9030B.

16.1.2. EPA 600, Methods for Chemical Analysis of Waters and Wastes, Sulfide (Titrimetric, Iodine), Method 376.1

16.1.3. TestAmerica North Canton Laboratory Quality Manual (LQM), current version

16.1.4. TestAmerica Quality Management Plan (QMP), current version

16.1.5. SW846, Test Methods of Evaluating Solid Waste, Third Edition, Titrimetric Procedure for Acid-soluble and Acid-insoluble Sulfides, Method 9034

16.1.6. Standard Methods, 18th Edition, 1992, Iodometric Method 4500-S2-E

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021 and S-Q-003

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.6. Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The lower reporting limit for Methods 376.1 and 4500-S2-E is 1 mg/L for water. The reporting limit for Method 9030A acid-insoluble sulfide is 5 mg/L for water and 30 mg/kg for solid. The acid-soluble sulfide reporting limit is 1 mg/L for water and 30 mg/kg for solid.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method Deviations

17.2.1. The lab does not perform the distillation procedure described in Method SW846 9030A.

17.2.2. The laboratory uses one collection flask instead of two.

17.2.3. The laboratory does not titrate the sample in the original container as specified in Method 376.1.

TESTAMERICA NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: FLASHPOINT CLOSED CUP

(SUPERSEDES: Revision 1, Dated 09/25/03)

Reviewed by:	Technology Specialist	7/18/07 Date
Approved by:	Quality Assurance Manager	6/28/07 Date
Approved by:	Environmental Health and Safety Coordinator	6-28-07 Date
Approved by:	Laboratory Director	7/18/07 Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Flashpoint by Pensky-Martens closed cup tester in a variety of wastes and liquids. It is based on ASTM D93-90 and SW846 Method 1010. Although the approximate working range is 20-200 °F, the test is generally considered complete when the temperature reaches 180°F without a measurable Flashpoint.

1.2. The associated LIMS method codes are AE (Method 1010) and GG (Method D93-90-Waste Matrix only).

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. The sample is heated at a slow constant rate with continual stirring if it is a liquid or water. A small test element is directed into the sample cup at regular intervals. The Flashpoint is the lowest temperature at which the test element causes the vapor above the sample to ignite.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Not Applicable

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, the facility addendum to the CSM, and this document.

5.2. 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
p-Xylene	Flammable Irritant	100 ppm-TWA	Inhalation of vapors may be irritating to the nose and throat. Inhalation of high concentrations may result in nausea, vomiting, headache, ringing in the ears, and severe breathing difficulties, which may be delayed in onset. High vapor concentrations are anesthetic and central nervous system depressants. Skin contact results in loss of natural oils and often results in a characteristic dermatitis. May be absorbed through the skin. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves MUST be worn when doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation, where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica North Canton associate. The situation must be reported **immediately** to a Laboratory Supervisor and the EH&S Coordinator.

5.9. In the event a sample ignites in the test apparatus do not attempt to remove the sample. Turn off the apparatus and flame. The flame 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.

5.10. When testing a sample, the analyst shall remain within eyesight of the flash point tester and will manually shut down the tester if it fails to automatically shut down following ignition.

6. EQUIPMENT AND SUPPLIES

6.1. Ignitor and detector

6.2. Pensky-Martens closed cup tester with stirrer and stirring motor

6.3. Flash point sample cup

6.4. Thermometer: 20 - 200°F range (made for the Pensky-Martens) or Thermocouple

6.5. Barometer

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. p-Xylene

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in glass containers at 4°C ± 2°C. Samples are not to be stored in plastic containers since volatile materials may diffuse through the walls of the enclosure.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS and Method Blank) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction

and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Duplicates

9.2.1. A sample duplicate (DU) is a second aliquot of an environmental sample, taken from the same sample container when possible, that is processed with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The sample and DU results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample/DU precision results are not necessarily representative of the precision for other samples in the batch.

9.2.2. Sample duplicates are performed at a minimum frequency of 10% per matrix (or one per analytical batch) and must meet laboratory-specific limits for precision. Soil and Waste matrices may be combined for batching purposes.

9.3. Control Limits

9.3.1. Control limits are established by the laboratory as described in SOP, NC-QA-0018.

9.3.2. Laboratory control limits are internally generated and updated periodically unless method specified. The latest version is easily accessible via the LIMs (QC Browser program).

9.4. Nonconformance and Corrective Action

9.4.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration of tester

10.1.1. Determine the flash point of p-xylene following the procedures outlined in 11.4

10.1.2. The tester is operating properly when a value of 81± 2°F is obtained.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo and is approved by a Technical Specialist and QA Manager. The Non-Conformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation Procedure

11.3.1. Not Applicable

11.4. Sample Analysis Procedure

11.4.1. Operate the Pensky-Martens tester according to the manufacturer's specifications, in a well-ventilated area away from flammable materials and significant air movement. Operating manuals are located in the laboratory near the tester. Performing this analysis under a hood is the best approach - ensure that the air intake and hood lights are turned off. Fill the sample cup to the designated line with sample and assemble the tester as directed.

11.4.1.1. Record the barometric pressure in cm.

11.4.2. A flash check must be analyzed with each analytical batch of samples. The compound p-Xylene is used to provide the analyst a reference true flash. The flash temperature is recorded on the analytical logsheet. The true flash temperature for p-Xylene is 81°F.

11.4.3. Pour the sample into the cup and record the initial temperature. If sample flashes, record the temperature at which the flash occurred on the analytical logsheet. Repeat sample for confirmation. If the temperature reaches 180°F and no flash occurs (see manual), turn off flashpoint and record > 180°F as the Flashpoint on the analytical logsheet.

11.4.4. If solvents are used to clean sample cups, be sure to clean thoroughly with reagent water to prevent contamination.

NOTE: Some samples may burn, but not flash. Record the initial temperature at which it burns and reanalyze for confirmation. Note this as a footnote when reporting.

Some samples have a Flashpoint below room temperature. If this is known, the sample should be chilled to just above freezing and then analyzed promptly to confirm a true Flashpoint.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logsheet, which may be in electronic format, including the analytical data from standards, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. If the sample did not flash, report > 180°F

12.2. Use the following equation to calculate the flashpoint:

$$\textbf{Flash} = F + [0.6\,(76 - (BP \times 2.54))]$$

Where: F = Observed Flash

BP = Barometric Pressure in cm

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications

13.2.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Refer to the Laboratory Sample and Waste Disposal plan.

15.2. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica North Canton. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.3. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.4. Waste Streams Produced by the method

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

15.4.1.1. **Solid samples.** Solids are put into the red can for the debris waste stream

15.4.1.2. **Liquid samples and waste solvents** Flammable wastes including the xylene standard are disposed of in the solvent waste stream located in the red can in the hood. Adding water to the solvent waste stream should be avoided.

16. REFERENCES

16.1. References

16.1.1. Annual Book of ASTM Standards, ASTM Method D93-90, Flashpoint by Pensky-Martens Closed Tester.

16.1.2. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Pensky-Martens Closed Cup Method for Determining Ignitability, Method 1010

16.1.3. Corporate Quality Management Plan (QMP), current version.

16.1.4. TestAmerica Laboratory Quality Manual (LQM), current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.5. Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Not Applicable

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica North Canton

SOP No. NC-MT-0010, Rev. 1.3
Effective Date: 04/23/07
Cover Page

Title: Hardness by Calculation
[Methods: SM2340B, SW846 Method 6010B]

Approvals (Signature/Date):

Roger K. Foos	12-14-07		12-14-07
Technology Specialist	Date	Health & Safety Coordinator	Date
Dorothy J. Lewson	12/13/07		12/14/07
Quality Assurance Manager	Date	Laboratory Director	Date

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STL North Canton
STANDARD OPERATING PROCEDURE

TITLE: HARDNESS BY CALCULATION

(SUPERSEDES: Rev. 1.2, Dated 03/01/05)

Reviewed by:	Roger K. Jones Technology Specialist	5-17-07 Date
Approved by:	Timothy J. Lewis Quality Assurance Manager	5/24/07 Date
Approved by:	William J. Deibel Environmental Health and Safety Coordinator	4-24-07 Date
Approved by:	Laboratory Director	5/17/07 Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis total hardness in water samples by SM2340B. The determination of calcium and magnesium concentrations in solution is achieved by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B.

1.2. The associated QuantIMS method code is SK.

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. This method describes the determination of total hardness by a calculation of the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter (mg/L).

2.2. Refer to the appropriate SOPs for details on sample preparation and analysis methods.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

3.2. Total hardness: The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter (mg/L), in a water sample.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. See the ICP analysis SOP for a discussion of physical, chemical, and spectral interferences that may affect calcium and magnesium concentration determinations.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document

5.2. 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.

5.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to ICP SOP, CORP-MT-0001NC, latest version

6.2. Refer to Inorganic Preparation SOP, CORP-IP-0003NC, latest version

7. REAGENTS AND STANDARDS

7.1. Refer to ICP SOP, CORP-MT-0001NC, latest version

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Preservation must be verified prior to analysis

9. QUALITY CONTROL

9.1. Refer to ICP SOP, CORP-MT-0001NC, latest version, for details on initial demonstrations, Inter-element Corrections (IECs), Rinse Time Determination, Background Correction Points, and Linear Range Verification (LR).

9.2. Batch Definition

9.2.1. A batch is a group of no greater than 20 samples. No quality control samples are processed with the batch since this is a calculation.

9.3. Nonconformance and Corrective Action

9.3.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to ICP SOP, CORP-MT-0001NC for details.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Refer to Inorganic Preparation SOP, CORP-IP-0003NC for details.

11.4. Sample Analysis

11.4.1. Refer to ICP SOP, CORP-MT-0001NC

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Total Hardness, mg equivalent CaCO3/L, = 2.497 (Ca, mg/L) + 4.118 (Mg, mg/L)

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file.

13.2. Training Qualifications:

13.2.1. The Group/Team Leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste streams produced by the method

15.2.1. No waste should be generated by this method (calculation only).

16. REFERENCES

16.1. References

16.1.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.1.2. CORP-MT-0001NC, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 6010B and 200.7.

16.1.3. CORP-IP-0003NC, Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods.

16.1.4. Standard Methods for the Examination of Water and Waste Water, 20th Edition, Method 2340B

16.1.5. Corporate Quality Management Plan (QMP), current version.

16.1.6. STL Laboratory Quality Manual (LQM), current version.

16.1.7. STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.3. Method Detection Limits and Instrument Detection Limits, S-Q-003 and NC-QA-0021

16.2.4. Supplemental Practices for DoD Project Work, NC-QA-0016

16.2.5. Standards and Reagents, NC-QA-0017

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. The reporting limit is 33 mg/L

17.2. Method deviations

17.2.1. Refer to ICP SOP, CORP-MT-0001NC, and Inorganic Preparation SOP, CORP-IP-0003NC, current versions

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Implementation Date: 6/28/05

SOP No: NC-WC-0084
Revision No: 5
Revision Date: 05/26/05
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STL STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY

(SUPERSEDES: REVISION 4, REVISION DATE 11/06/04)

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1. SCOPE AND APPLICATION

1.1. This method covers the determination of fluoride, chloride, nitrite, bromide, nitrate, ortho-phosphate and sulfate in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction Section 11.9) and leachates (when no acetic acid is used). This SOP is based on Method 300.0A and 9056A.

1.2. A listing of associated LIMs method codes is located in Section 8.2.

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A 25 uL volume of sample is introduced into the ion chromatograph. The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn or guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppresser column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2. The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of concentrated eluent to each standard and sample.

4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

4.4. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5. The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

5.2. Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.3. 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
Potassium Nitrate	Oxidizer	None	Causes irritation to the respiratory tract, skin and eyes. Symptoms may include coughing, shortness of breath. Symptoms include redness, itching, and pain.

Sodium Fluoride	Poison	2.5 Mg/M3-TWA as F	**Highly Toxic.** Causes severe irritation to the respiratory tract, symptoms may include coughing, sore throat, and labored breathing. Causes irritation, with redness and pain. Solutions are corrosive. Eye irritant! May cause irritation and serious eye damage. Effects may not appear immediately.
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.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore; unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation when possible. All samples with a pink stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S co-ordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to the EH&S Coordinator and to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2. Ion Chromatograph -- Analytical system complete with ion chromatograph and all required accessories including analytical columns, compressed gases and detectors.

6.2.1. Anion guard column: A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with same substrate as the separator column. 4 x 50 mm, Dionex IonPac AG14 P/N 46134, or equivalent.

6.2.2. Anion separator column: The separation shown in Figure 1 was generated using a Dionex IonPac AS14 column (P/N 46134). Equivalent column may be used if comparable resolution is obtained, and the requirements of Sect. 9.2 can be met.

6.2.3. Anion suppresser device: Dionex anion micro membrane suppresser (P/N 37106) or ASRS-Ultra Self-Regenerating Suppressor (4mm) P/N 53946 or equivalent.

6.2.4. Detector -- Conductivity cell: approximately 1.25 uL internal volume, Dionex, or equivalent.

6.2.5. Dionex –PeakNet 5.1Data Chromatography Software or equivalent.

6.3. Assorted laboratory glassware (pipettes, volumetric flasks, etc.).

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2. Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.3. Eluent solution: sodium bicarbonate (CASRN 144-55-8) 1.0 mM, sodium carbonate (CASRN 497-19-8) 3.5 mM. Dissolve 1.680 g sodium bicarbonate ($NaHCO_3$) and 7.417 g of sodium carbonate (Na_2CO_3) in reagent water (7.2) and dilute to 100 mL in a volumetric flask. Take 10 mL of this concentrated eluent solution and dilute to 2 L for use as the working eluent solution or dissolve the entire bicarbonate/carbonate amount in 20 L of reagent water.

7.4. Stock solutions (1,000 mg/L): All stocks are purchased from commercial sources. Primary and secondary sources are required for each target analyte.

7.4.1. Commercial stock solution A: F^- - 25 mg/L, Cl^- - 500 mg/L, Br^-- 100 mg/L, NO_3^- - N- 25 mg/L, PO_4 – P - 25 mg/L, SO_4^{-2}- 500 mg/L. The stock solution may also be at the folloing concentrations: F^- - 125 mg/L, Cl^- - 2500 mg/L, Br^-- 500 mg/L, NO_3^- - N- 125 mg/L, PO_4 – P - 125 mg/L, SO_4^{-2}- 2500 mg/L.

7.4.2. Commercial stock solution B: NO_2^- - N- 25 mg/L. The spike solution may also be at 125 mg/L.

7.4.3. Commercial IC Spike solution A: : F^- - 125 mg/L, Cl^- - 2500 mg/L, Br^-- 500 mg/L, NO_3^- - N- 125 mg/L, PO_4 – P - 125 mg/L, SO_4^{-2}- 2500 mg/L

7.4.4. Commercial IC Spike solution B: NO_2^- - N- 125 mg/L

7.5. Working standards: Prepare calibration standard #5 in a 10 mL volumetric flask and transfer to a vial. Adjust the amount of stock solution used to prepare the working standards if the stock concentration differs from 1000 mg/L as assumed Alternatively prepare Cal standard #5 by mixing 4.0 mL commercial stock A, 4.0 mL commercial stock B and 2.0 mL of eluent or, if the more concentrated solution is used, by mixing 0.8 mL A, 0.8 mL B, and 8.4 mL of eluent.

Calibration Standard #5

Analyte	mL of Stock	Final Conc.
Fluoride	0.10mL	10.0 mg/L
Chloride	2.0 mL	200. mg/L
Nitrite	0.10 mL	10.0 mg/L
Bromide	0.40 mL	40.0 mg/L
Nitrate	0.10 mL	10.0 mg/L
Ortho-Phosphate	0.10 mL	10.0 mg/L
Sulfate	2.0 mL	200. mg/L

7.5.1. In 5 mL PolyVials prepare the following calibration standards in reagent grade water. Final concentrations of working standards are shown below.

Calibration Standard #4: take 2.50 mL of calibration standard #5 and add 2.50 mL of eluent.

Calibration Standard #2: take 250 µL of calibration standard #5 and add 4.75 mL of eluent.

Calibration Standard #1: take 25.0 µL of calibration standard #5 and add 4.975 mL of eluent.

Calibration Standard #3: take 1.25 mL of calibration standard #5 and add 3.75 mL of eluent.

Calibration Standard #1

Analyte	25.0 µL of Cal Std #5	Final Conc
Fluoride		0.05 mg/L
Chloride		1.0 mg/L
Nitrite		0.05 mg/L
Bromide		0.20 mg/L
Nitrate		0.05 mg/L
Ortho-Phosphate		0.05 mg/L
Sulfate		1.0 mg/L

Calibration Standard #2

Analyte	250 µL of Cal Std #5	Final Conc.
Fluoride		0.5 mg/L
Chloride		10. mg/L
Nitrite		0.5 mg/L
Bromide		2.0 mg/L
Nitrate		0.5 mg/L
Ortho-Phosphate		0.5 mg/L
Sulfate		10. mg/L

Calibration Standard #3

Analyte	1.25 mL of Cal Std #5	Final Conc.
Fluoride		2.5 mg/L
Chloride		50. mg/L
Nitrite		2.5 mg/L
Bromide		10. mg/L
Nitrate		2.5 mg/L
Ortho-Phosphate		2.5 mg/L
Sulfate		50. mg/L

Calibration Standard #4

Analyte	2.5 mL of Cal Std #5	Final Conc.
Fluoride		5.0 mg/L
Chloride		100 mg/L
Nitrite		5.0 mg/L
Bromide		40. mg/L
Nitrate		5.0 mg/L
Ortho-Phosphate		5.0 mg/L
Sulfate		100 mg/L

7.5.2. Prepare or purchase a secondary stock standard(s) using a standards source other than that used for the primary standards as described in Section 7.5. Dilute these stock standards to as indicated in the table below to prepare the mixture to be used for the LCS and CCV solution. The CCV/LCS solution may be prepared by mixing 20 mL of A and 20 mL of B and diluting to 200 mL. A higher dilution solution may also be prepared by taking 5mL of A (5X) and 4 mL of B (5X) and diluting to 200 mL.

LCS & Continuing Calibration Verification Solution

Analyte	Final Conc. (V_f=5ml)
Fluoride	2.5 mg/L
Chloride	50. mg/L
Nitrite	2.5 mg/L
Bromide	10. mg/L
Nitrate	2.5 mg/L
Ortho Phosphate	2.5 mg/L
Sulfate	50. mg/L

7.5.3. Prepare or purchase a secondary stock standard(s) using a standards source other than that used for the primary standards as described in Section 7.5. Dilute these stock standards to prepare the mixture to be used for the Matrix Spike solution. Alternatively purchase these mixes (ready to use) from a commercial source. Add 100 uL of each IC Spike solution to 5 mL of sample when preparing the MS. Dilute as needed after spiking the sample.

Matrix Spike "True" Values

Analyte	Final Conc.
Fluoride	2.5 mg/L
Chloride	50. mg/L
Nitrite	2.5 mg/L
Bromide	10. mg/L
Nitrate	2.5 mg/L
Ortho Phosphate	2.5 mg/L
Sulfate	50. mg/L

NOTE: Stock standards, calibration standard #5 and LCS standard should be stored in the dark at 4° ± 2°C. Replace these standards when instrument response indicates target analyte degradation may have occurred or after the standard has expired (12 months commercial mix or

6 months in house mix), which ever occurs first. Nitrite and ortho-phosphate are particularly light and oxygen sensitive.

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.2. Sample preservation and holding times for the anions that can be determined by this method for water samples are as follows:

QuanTIMs Method Code		Analyte	Preservation	Holding Time
EPA 300.0A	SW846 9056A	Fluoride	4° ± 2°C	28 days
C8	3C			
CX	3D	Chloride	4° ± 2°C	28 days
GO	3E	Nitrite	4° ± 2°C	48 hours
GM	3F	Bromide	4° ± 2°C	28 days
C9	3G	Nitrate	4° ± 2°C	48 hours
DO	3H	Ortho Phosphate	4° ± 2°C	48 hours
CY	3I	Sulfate	4° ± 2°C	28 days

Note: Soil leachates will follow the same preservation and holding times as the water samples; starting from the time of extraction.

9. QUALITY CONTROL

9.1. The STL QC Program document provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

9.2. Table I provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.3. Initial Demonstration of Capability

9.3.1. Prior to the analysis of any samples by Ion Chromatography, the following requirements must be meet:

9.3.1.1. Method Detection Limit (MDL): An MDL must be determined prior to analysis of any samples. The MDL is determined using seven replicates of eluent spiked with the anions of interest that has been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis. The spike level must be greater than the calculated MDL but less than or equal to 10x the MDL. The result of the MDL determination must be below the STL reporting limit.

9.4. Batch definition: Preparation and QC batch definitions are provided in the STL QC Policy.

9.5. Method Blank (MB): One method blank must be processed with each preparation batch. The method blank consists of eluent that has been taken through the entire preparation and analytical process. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest above the reporting limit.

9.6. Laboratory Control Sample (LCS): One LCS must be processed with each preparation batch and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. If the result is outside established control limits the system is out of control and corrective action must occur. Until in-house limits are established, a control limit of 90 - 110% recovery must be applied. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable. The LCS is prepared from a separate stock standard, or neat material, of a different manufacturer than the stock, or neat material, used to prepare the calibration standard.

9.7. Matrix Spike/Matrix Spike Duplicate (MS): One MS sample must be analyzed every 10 samples. A matrix spike (MS) is a field sample to which a known concentration of target analyte has been added. Some client specific DQO's may require the use of sample duplicates in place of or in addition to MS's. The MS result is used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample,

these results may have immediate bearing only on the specific sample spiked. Spiking levels will be the same as the LCS values.

- If the MS recovery or RPD falls outside the acceptance range, the recovery of the analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 80-120% recovery and 20% RPD must be applied to the MS.
- If the native analyte concentration in the MS exceeds 4x the spike level for that analyte, the recovery data is reported as "amount" MSB. The Exception Code is changed to NC. The following two footnotes will appear on the report page 'NC The recovery and/or RPD were not calculated." "MSB The recovery and RPD were not calculated because the sample amount was greater than four times the spike amount."
- If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted.
- If the recovery of the LCS is outside the limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.
- If an MS is not possible due to limited sample volume then a LCS duplicate must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike limits.

9.8. Continuing Calibration Verification (CCV/CCB): Continuing calibration is verified by analyzing the calibration standard after every ten (10) samples . The CCV must fall within +/- 10% of the true value for each target analyte. A CCB is analyzed immediately following the CCV to monitor low level accuracy and system cleanliness. The CCB result must be below the reporting limit for that analyte. If either the CCV or CCB fail to meet criteria, the analysis must be terminated, the problem corrected and repreparation and analysis of all samples following the last CCV and CCB which were in control.

9.9. Reporting Limit Check – Drinking Water Samples. A reporting limit check must be analyzed every day drinking water samples are analyzed. The acceptance criteria is ± 30% for the check.

9.10. Control Limits

9.10.1. Control limits are specified in the method.

9.10.2. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.11. Method Detection Limits (MDLs) and MDL Checks

9.11.1. MDLs and MDL Checks are established by the laboratory as described in SOPs S-Q-003 and NC-QA-0021.

9.11.2. MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.12. Nonconformance and Corrective Action

9.12.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Establish ion chromatographic operating parameters equivalent to those indicated in table 2. Refer to Table 3 for typical standard run retention times. Other than the presence of the analytical column the instrument conditions are the same.

10.2. For each analyte of interest, prepare a **minimum** of 3 calibration standards and a blank by adding accurately measured volumes of one or more stock standards to a volumetric flask and dilution to volume with eluent. If a sample analyte concentration exceeds the calibration range the sample may be diluted to fall within the range. If this is not possible then three new calibration concentrations must be chosen, two of which must bracket the concentration of the sample analyte of interest. Each attenuation range of the instrument used to analyze a sample must be calibrated individually.

10.3. Using an injection volume of 25 uL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded. All analytes will be calibrated using a quadratic regression forced through the origin. Correlation coefficients (R^2) must be 0.995 or better.

10.3.1. For Drinking Water samples, a linear curve, that is not forced through the origin, is used.

10.3. Initial Calibration Verification (ICV) – The Initial Calibration is verified at the start of each day following calibrating prior to sample analysis. The acceptance criteria is ± 10% of the true value. If this criteria is not met, the instrument must be re-calibrated.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance

Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Table 2 summarizes the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Sect. 9.2 are met.

11.4. Check system calibration daily as outlined in Table 1 and, if required, recalibrate as described in Sect 10.

11.5. Load and inject a fixed amount (25 uL) of settled & filtered sample. If the sample is cloudy then it should be filtered prior to loading into the autosampler polyvial. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

11.6. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of various concentration. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms since retention time is concentration dependent for most analytes..

11.7. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of eluent and reanalyze.

11.8. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

NOTE: Retention time is affected by concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. See Table 3. In some cases this peak migration may produce poor resolution or identification.

11.9. The following extraction should be used for solid materials: Add an amount of reagent water equal to ten times the weight of dry solid material taken as a sample. This slurry is mixed for one hour using a magnetic stirring device or tumbler. Filter the resulting slurry before injecting using a 0.45 um membrane type filter. This can be the type that attaches directly to the end of the syringe

11.10. Should more complete resolution be needed between peaks the eluent (7.3) can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

11.11. Analytical Documentation

11.11.1. Record all analytical information in the analytical logbook/logsheet which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MSs, and any corrective actions or modifications to the method.

11.11.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.11.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.11.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Prepare a calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.

12.2. Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12.3. Report results in mg/L for aqueous samples and mg/Kg for 1 hour leachates and mg/L for 18 hour leachates of solid samples.

12.4. Report NO_2^- as N, NO_3^- as N, $HPO_4^=$ as P

13. METHOD PERFORMANCE

13.1. The reporting limits for the following analytes are based on a 25 uL injection volume:

Analyte	Water RL	Soil RL
Fluoride	1.0 mg/L	10 mg/kg
Chloride	1.0 mg/L	10 mg/kg
Nitrite	0.1 mg/L	5 mg/kg
Bromide	0.1 mg/L	5 mg/kg
Nitrate	0.1 mg/L	5 mg/kg
O-Phosphate	0.5 mg/L	5 mg/kg
Sulfate	1.0 mg/L	10 mg/kg

13.2. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. The analyst must be given two blind performance samples to analyze or process for analysis. Upon successful completion of the performance evaluation (PE) samples, these analyses will be documented as initial qualification,. Requalification must be performed annually thereafter for this procedure. The group/team leader must document the training and PE performance and submit the results to the QA Manager for inclusion in the associate's training files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

15.2. Waste Streams Produced by the Method

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

15.2.1.1. Spent samples. Solid samples are disposed of as solid debris waste in the container labeled "Solid Waste."

15.2.1.2. Alkaline and/or acidic waste generated by the analysis. Aqueous waste can be poured down the drain if the pH is between 4 and 10. Any sample waste generated that is not in this pH range must be collected and disposed of in the designated acid waste drum located in the lab. This waste is collected in the laboratory in a designated container identified as "Acid Waste".

15.2.1.3. Contaminated plastic materials such as IC syringes, filters, caps and vials utilized for sample preparation. This waste is disposed of in containers labeled "Solid Waste."

16. REFERENCES

16.1. References

16.1.1. Method 300.0, "Determination of Inorganic Anions by Ion Chromatography", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, Revision 2.1, August 1993.

16.1.2. Method 9056A, "Determination of Inorganic Anions by Ion Chromatography", SW846, Test Methods for Evaluating Solid Waste, Third Edition, Draft Revision 1, September 1999.

16.1.3. STL North Canton Laboratory Quality Manual (LQM), current version.

16.1.4. Corporate Quality Management Plan (QMP), current version.

16.1.5. STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021 and S-Q-003.

16.2.5. Navy/Army SOP, NC-QA-0016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Attachment #1, method Flow Chart

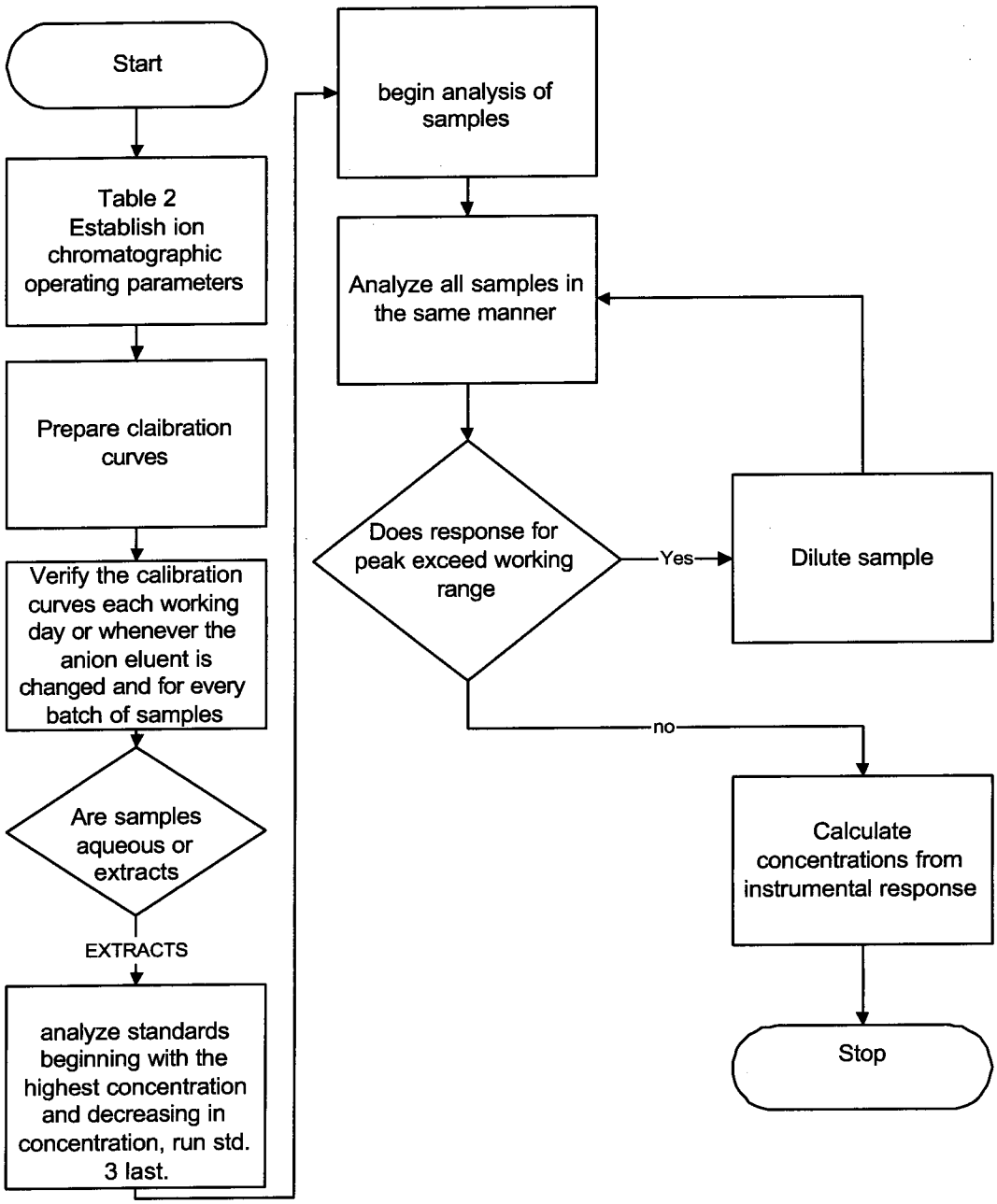
17.3. Table 1, Quality Control Samples

17.4. Table 2, Standard Instrument Operating Parameters

17.5. Table 3, Retention Time Matrix

17.6. Figure 1, Example Chromatogram

Determination Of Inorganic Anions By Ion Chromatography



ATTACHMENT #1

TABLE 1

QUALITY CONTROL SAMPLES

QC Samples	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Verification (ICV)	At the start of each day following calibrating prior to sample analysis	+/- 10% of true value	Recalibrate and reanalyze
Initial Calibration Blank (ICB)	After Initial Calibration Verification and prior to sample analysis	< the Reporting Limit	Reprepare and reanalyze
Laboratory Control Sample (LCS)	1 per batch of 20 samples	Meets laboratory historical limits	Reanalyze all samples associated with unacceptable LCS
Matrix Spike Sample (MS)	1 MS per every 10 samples	Meets laboratory historical limits	Supervisor's technical judgment
Continuing Calibration Verification (CCV)	Between each group of 10 injections and at the end of the analytical sequence	+/- 10% of true value	Recalibrate and reanalyze all samples since the last acceptable CCV
Continuing Calibration Blank (CCB)	Between each group of 10 injections and at the end of the analytical sequence	< the Reporting Limit	Recalibrate and reanalyze all samples since the last acceptable CCB

TABLE 2

Standard Instrument Operating Parameters

Standard Conditions:

Eluent Pump Rate:	1.20 mL/min (DX-120 and DX-320)
Sample Loop:	25 uL
Eluent:	1.0mM sodium bicarbonate, 3.5mM sodium carbonate
Detector output	Baseline conductivity should be 15 - 20 uS prior to sample analysis.

TABLE 3

Standard Run Retention Time Matrix (minutes)*

	Concentration (mg/L)													
Analyte	0.05	0.2	0.5	1	2	2.5	5	10	20	40	50	100	200	RT window
F^-	2.75		2.75			2.75	2.75	2.75						
Cl^-				3.97				3.98			4.03	4.08	4.17	
NO_2^-	4.80		4.80			4.78	4.78	4.80						
Br^-		6.15			6.13			6.10	6.08	6.07				
NO_3^-	7.33		7.27			7.17	7.13	7.07						
$o\text{-}PO_4^{2-}$	9.53		9.53			9.52	9.50	9.48						
SO_4^{2-}				11.50				11.48			11.43	11.38	11.27	

* Analyte retention time is concentration dependent for most anions. Retention time increases with increasing concentration for chloride. Retention time decreases with increasing concentration for bromide, nitrate, ortho-phosphate and sulfate.

EXAMPLE ION CHROMATOGRAM

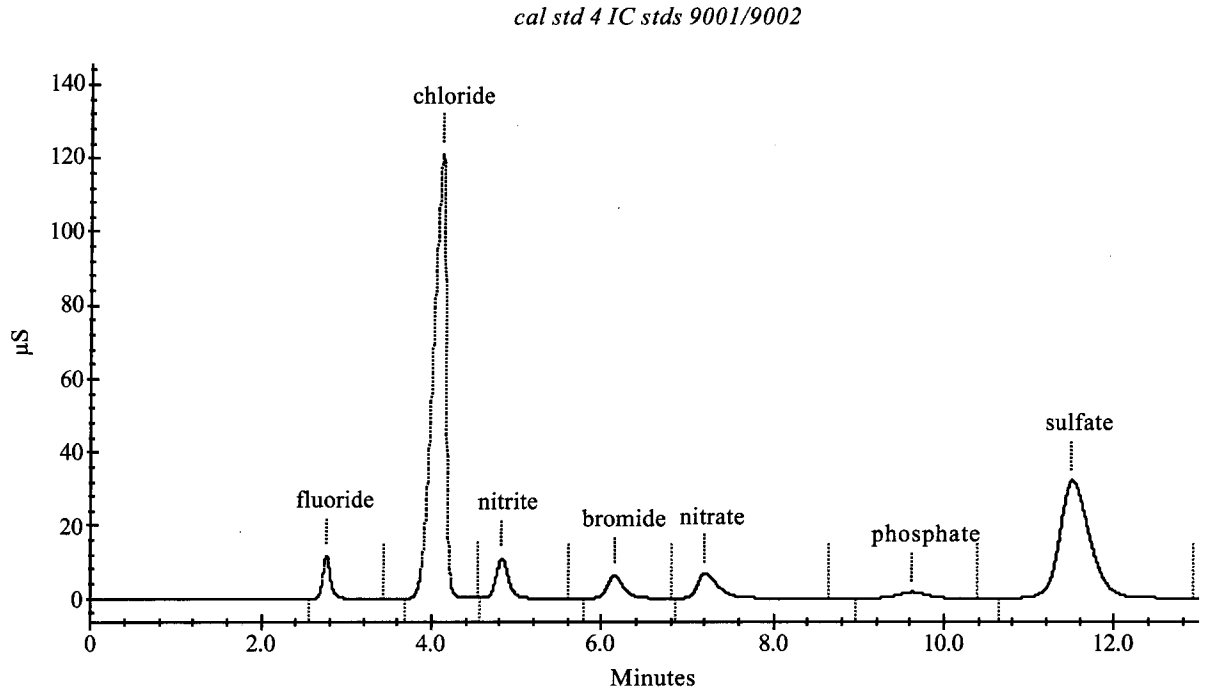


Figure #1

Title: RELEASING and CLEANING of SAMPLE CANISTERS and CLEANING, CALIBRATION, and SETTING of FLOW REGULATORS and VACUUM GAUGES

Approvals (Signature/Date):

3/4/08	03/14/2008
Steven Gonzales Sample Control Supervisor Date	William Nash Environmental Health & Safety Coordinator Date
3-18-2008	3/18/08
Maria Friedman Quality Assurance Manager Date	Elizabeth Winger Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This document describes the procedure that ensures all passivated canisters are properly released, cleaned, and certified to less than 0.20 ppbv of target analytes or to less than the method detection limit (MDL), or to levels that meet client-specific requirements.

1.2. This document also discusses the procedures for cleaning, calibrating, and setting flow regulators and vacuum gauges.

1.3. Once a sample in a passivated canister has been analyzed for all requested parameters, the canister is cleaned/certified and made available for collecting new samples.

1.4. Cleaning also involves the identification and repair of any damaged canisters.

1.5. It is TestAmerica Los Angeles' policy to release passivated canisters for cleaning seven (7) days after mailing the final report. However, contractual requirements may dictate a longer holding time before release. This information must be received in writing from the client.

2. SUMMARY OF METHOD

2.1. Releasing Canisters

2.1.1. A passivated canister exists in the laboratory in one of six states.

2.1.1.1. State 1: Awaiting analysis

2.1.1.2. State 2: Analysis in progress

2.1.1.3. State 3: Available for release

2.1.1.4. State 4: Available for cleaning

2.1.1.5. State 5: Cleaned and awaiting certification

2.1.1.6. State 6: Cleaned and certified

NOTE: This standard operating procedure (SOP) describes the process by which a canister advances from state 3 to state 6.

2.2. Canister Cleaning

2.2.1. TestAmerica Los Angeles has two automated canister cleaning systems that are capable of processing 12 canisters per batch per system.

2.2.2. A batch of heated canisters are cycled through a process of evacuating to a vacuum of approximately 25 inches of mercury and then pressurizing to approximately 30 pounds per square inch gauge (psig) using humidified, ultra high purity (UHP) nitrogen.

2.2.3. The number of cycles is determined by the level of contamination of the dirtiest canister in the batch - the "screen can". The level of contamination is typically determined by the results of the EPA TO-14A or EPA TO-15 analysis of the air sample in the canister.

2.2.4. Upon completion of the appropriate cycles, the screen can is analyzed by EPA TO-14A or EPA TO-15 to confirm that there are no target analytes above 0.20 ppbv, the MDL, or the client-specific requirements. This process is known as "certification".

2.2.5. Once certification has been performed, the canisters in the batch are evacuated to a final vacuum of $\leq$ 0.050 torr and may then be shipped to clients.

3. DEFINITIONS

3.1. Passivated canister: Commonly referred to as SUMMA canister, SilcoCan, or T.O.-Can in 1.0-Liter, 6-Liter, and 15-Liter sizes.

3.1.1. SUMMA Canister: A spherical stainless steel container, which interior has been specially treated by a process (SUMMA passivation) that renders all surfaces inert to volatile organic compounds (VOCs).

3.1.2. SilcoCan: A sampling canister manufactured by Restek Corporation using the Restek Silcosteel® process to coat the interior of the canister with fused silica, rendering it inactive to most VOCs.

3.1.3. T.O.-Can: A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary electropolishing process and extensively cleaned using an ultrasonic method that ensures a high-quality, passivated surface that maintains the stability of VOCs during storage.

3.2. Vacuum Flow Regulator (VFR): A device which, when connected to a passivated canister, regulates the flow of sample into the canister so that a timed, representative sample can be obtained (also called a composite sample), as opposed to an unregulated, instantaneous sample (grab sample).

3.3. Particulate Filter: A cylindrical stainless steel fitting containing a fritted metal disc, which is connected to the valve of a passivated canister or VFR, to prevent particulate matter from entering and damaging the canister or VFR.

3.4. Pressure Gauge: Device used to measure the vacuum or pressure in a passivated canister. Units of measure range from 30 to 0 inches of mercury (for vacuum) to 0 to 30 psig (for positive pressure). All pressure units are converted to pounds per square inch absolute (psia).

3.5. Canister certification: The process by which passivated canisters are analyzed and confirmed to be clean to less than 0.20 ppbv of each target analyte or to less than the MDL, or to the client-specific requirements.

3.6. Screen can: The canister in a cleaning batch that has been identified as the most contaminated and which undergoes the certification process.

3.7. Batch: A group of 12 canisters cleaned at the same time and on the same manifold.

3.8. Released: The disposition of a passivated canister.

3.9. Equipment Rental Record (ERR): The record that documents which equipment was rented to a client.

4. INTERFERENCES

4.1. The canisters can be contaminated during the cleaning process if the nitrogen cylinders used during the humidified pressurization step become empty.

4.2. Contamination can also occur if the water used for humidification is dirty. Only distilled or purge-and-trap grade water must be used.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

5.2. Specific Safety Concerns or Requirements

5.2.1. Canisters under pressure must be handled with care and should never be pressurized over 40 psig.

5.2.2. Heating bands used for canister cleaning may be hot – use care when handling.

5.2.3. Liquid Nitrogen can burn. Use Cryogloves when handling cylinders.

5.2.4. All compressed gas cylinders must be securely retained with the regulator covers in place when not in use.

5.2.5. The exhaust from vacuum pumps used in the evacuation of contaminated canisters and flow controllers must be vented to a

properly functioning fume hood. If the exhaust is not vented directly to a fume hood, prior to attaching the canisters or flow regulators to the cleaning manifold, vent the canisters or flow controllers directly into a properly functioning fume hood.

5.2.6. Temperature appropriate gloves must be worn when working with hot or cold items.

5.3. Primary Materials Used

5.3.1. None.

6. EQUIPMENT AND SUPPLIES

6.1. Entech 3100+ automated canister cleaning system with 12-canister manifold

6.2. Regulator for UHP nitrogen-single-staged, stainless steel diaphragm (Scott-51-08CS-CGA)

6.3. Vacuum pump (Edwards Model E2M8)

6.4. Rough pumps (KNF Neuberger 726.3ANP, or equivalent)

6.5. Assorted stainless steel tubings, fittings, and valves

6.6. Pressure gauges capable of reading from a vacuum of 30 inches of mercury to 30 psig

6.7. Pirani Model 315 Micro controller

6.8. Varian valve - Model NW-25

7. REAGENTS AND STANDARDS

7.1. UHP nitrogen (used for canister cleaning and pressurization of the screen can)

7.2. Distilled or purge-and-trap grade water

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

8.1. Not applicable.

9. QUALITY CONTROL

9.1. A batch of canisters is controlled through the analysis of the screen can.

9.2. Upon completion of the cleaning cycles, the screen can is humidified and pressurized to 30 psig and submitted to the laboratory for EPA TO-14A or EPA TO-15 analysis.

9.3. The laboratory will analyze the screen can using EPA TO-14A or EPA TO-15 procedures. Results of the analysis are submitted to the Air Sample Control Technician and are signed and dated by the analyst.

9.4. In order to be certified, the screen can must not contain target analytes greater than or equal to the method criteria for the specified analytical method (in most cases, 0.20 ppbv or greater than the MDL). If both the laboratory and the client agreed upon different criteria, the certification will be based on that criteria. This will be noted on the certification paperwork and in the project records.

9.5. If the screen can does not pass the certification criteria, the entire batch is re-cleaned and the screen can undergoes analysis for a second time. If the screen can fails again, it will go through the cleaning and analysis procedure a third time.

9.6. If the screen can fails a third time, the entire batch of canisters is separated out and each canister is then individually screened. Those canisters that meet acceptance criteria can be used for sampling. Any canister that fails will be removed from service.

9.6.1. Once analyzed for the required method, the laboratory will return the screen can and paperwork to the Air Sample Control Technician. Any canister that did not pass criteria must be placed on the designated "Dirty" canister shelves for re-cleaning. Those screen canisters that passed criteria are placed on the "Screened" shelves along with the rest of the batch canisters.

9.7. If all criteria have been met, the Canister QC Certification sheet is completed listing the date cleaned, QC data file number, and all related canister ID numbers. See Appendix A.

9.8. The quantitation report and chromatogram of the screen can analysis are filed with the batch certification and are kept in the monthly certification file and with the ERR. A copy is also filed in the respective project folders.

9.9. Upon completion of certification, the batch of canisters undergoes a final evacuation down to <0.050 torr. These canisters are placed on the "Clean" shelves. Prior to submission of canisters for shipment, the vacuum is checked again to 30 inches of mercury.

10. PROCEDURE

10.1. Any variation in procedure shall be completely documented using a Nonconformance Memo (NCM) and is approved by a technical specialist and the Quality Assurance (QA) Manager. If contractually required, the client shall be

notified. The NCM shall be filed electronically and a copy kept in the project file. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described. If contractually required, the client shall be notified.

10.2. Calibration

10.2.1. The master gauge, flow meters, and pressure gauges are calibrated.

10.2.1.1. The master gauge is calibrated annually by a certified outside vendor. These records are maintained in the QA files.

10.2.1.2. The process flow meter, used in the laboratory area for preparing standards and in the Sample Control area to set the flow regulators at the client-requested flow rates for time-weighted sampling events, are checked and calibrated quarterly by the Air Sample Control Technician. These checks are documented in the process meter logbook.

10.3. Releasing Canisters

10.3.1. Data obtained from analysis performed on the passivated canister undergo a validation process consisting of three levels:

10.3.1.1. Level I - Bench-level or analyst review

10.3.1.2. Level II - Second-level or peer review

10.3.1.3. Level III - Project Manager (PM) review

10.3.2. After analysis, the analyst indicates on the canister tag the volume or amount of sample used. This information will later aid the Sample Control Technician in determining which canisters can be cleaned together in a batch. Likewise, it will facilitate identification of the dirtiest canister in the batch, which will be later called the "screen can".

10.3.3. The tagged and labeled canister is transferred to the storage area. It remains there until it has been cleared for release. Canisters may not be released until at least seven days have passed after mailing the final report. A summary of the lot numbers ready for release can be generated by the PM.

10.3.4. There can be no partial releases. Only whole projects can be released. For example, if a project of 20 canisters is received, they can only be cleaned when all 20 canisters have been released.

10.3.5. Clients may request the temporary archiving or holding of specific canisters within a batch. In this case, the other canisters may be released for cleaning.

10.4. Canister Cleaning - Automated system (see Figure 1)

10.4.1. Verify that the bubbler contains sufficient amount of purge-and-trap grade water.

10.4.2. Attach all canisters to the manifold arms. Attach all heating bands to the canisters.

10.4.3. Determine the number of cycles needed and adjust the controller to the appropriate setting. Verify that filling and pumping cycles are at 6-minute intervals each. The number of cycles depends upon the level of contamination in the dirtiest canister. A volume, (100 mL, 500 mL, etc.), is indicated on the canister tag by the analyst. This signifies the amount of sample used for analysis. The lower the volume, the higher the contamination. Generally, canisters which have volumes of 100 to 1000 mL require only 1-8 cycles; 10 - 100 mL require 16 cycles; 1 - 10 mL require 24 cycles; and volumes less than 1 mL requires overnight to a full weekend of cycles.

10.4.4. Initiate the canister cleaning cycle.

10.4.5. Upon completion of the cleaning cycle, perform the final evacuation of the canisters by initiating the rough pump until the pressure reading approaches 0.20 to 0.50 psia.

10.4.6. Close all canister valves. Put the canister cleaning system controller in the Standby mode. Detach the screen can, humidify with 50 uL of purge-and-trap water, and pressurize to 30 psig with UHP nitrogen. Submit the screen can with the batch information sheet to the laboratory for certification and analysis.

10.4.7. If the screen can fails the certification criteria, the batch of canisters associated with the screen can must be re-cleaned and re-certified until criteria are met (restart from section 10.4.1).

10.4.8. When the certification is complete, the entire batch should be evacuated to $\leq$ 0.050 torr.

10.5. Flow Regulator Cleaning

10.5.1. Upon receipt of the flow regulator, it is checked and placed in the "Dirty" or "Used" bin.

NOTE: If the flow regulator is contaminated with oil or water, it is red-tagged and sent out for service.

10.5.2. To start the cleaning process, open the flow regulator valve.

10.5.3. Attach flow regulator to the cleaning manifold.

10.5.4. Pass a steady flow of UHP nitrogen gas through the flow regulator.

10.5.4.1. For flow regulators with no attached pressure gauge, allow nitrogen to pass through for at least 10-15 minutes. For flow regulators with pressure gauge, allow nitrogen to pass through for least 10-15 minutes since the gas flow is more restricted.

10.5.5. Detach flow regulator from the manifold.

10.5.6. Check for leak.

10.5.7. Plug (close) the inlet valve of the flow regulator.

10.5.8. Attach flow regulator to the Leak Test canister.

10.5.9. Open the canister valve to the flow regulator.

10.5.10. Check if the canister vacuum gauge has the same reading as the flow regulator gauge.

10.5.11. Close the canister valve to the flow regulator.

10.5.12. Check the flow regulator gauge. If there is no change (decrease), then there is no leak. If there is a leak, red-tag the flow regulator and send it out for service.

10.5.13. The cleaned flow regulator is placed in the "Cleaned" bin and logged into the "cleaned" Canisters and Regulators Cleaning Logbook.

10.6. Flow Regulator – Calculations for setting flows

10.6.1. 0.25- to 12-hour composite

10.6.1.1. For a standard 6-Liter canister, the target fill volume is typically five liters (5000 mL). The upper flow rate is then calculated by dividing 5000 mL by the fill time in minutes. The lower flow rate is 95% of the upper rate.

Example: For a four-hour composite, the upper flow rate would be 5000 mL/240 minutes = 20.8 mL/min. The lower flow rate would be 95% of 20.8 mL/min, which is 19.8 mL/min. Setting the flow regulator between 19.8 and 20.8 mL/min should yield good results.

10.6.2. 24-hour composite

10.6.2.1. The standard flow regulators are designed for flows above 5 mL/min. Therefore, 24-hour composite samples require modifying the above criteria for best results. Typically, the

flow rate is set between 3.5 and 4.0 mL/min. Theoretically for a 24-hour composite, the 4.0 mL/min flow will fill a 6-liter canister with 5760 mL of sample for a final vacuum of 1.2 inches of mercury, and the 3.5 mL/min flow will fill the canister with 5040 mL at 4.8 inches of mercury. From the 5 inches of mercury differential pressure required for constant flow, it can be seen that setting the flow closer to the 3.5 mL/min limit is more desirable.

10.6.3. Higher Elevation

10.6.3.1. As the elevation at the point of sampling increases, the amount of atmospheric pressure available to fill a canister decreases. Flow rates should be lowered to take this into account and prevent the sample flow from going to zero before the sampling period is over.

NOTE: Flow rates should be decreased by about 4% for every 1000 feet of elevation above sea level.

Example: A four-hour composite in a 6-Liter canister would normally have a setting between 19.8 and 20.8 mL/min. If sampling is to occur at 3500 feet, the flow range should be adjusted down approximately 14%. This would give a setting between 17.0 and 17.9 mL/min.

10.7. Vacuum Gauge Cleaning

10.7.1. Vacuum (pressure) gauge is cleaned before it is sent out to the client.

10.7.2. Attach a length of disposable tubing (two-inch Tygon tube) to the UHP nitrogen source.

10.7.3. Flush the gauge, making sure that the pressure goes up to 30 psi each time. Repeat procedure 15 to 20 times.

10.7.4. Dispose of tubing. Pressure gauge is now ready for shipment.

10.8. Vacuum Gauge Calibration

10.8.1. Verify that the gauge reads 0 psig $\pm$ 0.25 at atmospheric pressure.

10.8.2. Verify gauge in vacuum.

10.8.2.1. Attach gauge to calibrating gauge canister (canister in vacuum with calibrated gauge and an attachment for the gauge to be calibrated).

10.8.2.2. Open canister valve to gauge to be calibrated.

10.8.2.3. Note gauge reading as compared to calibrated gauge. Acceptable range is ± 0.75 inch of mercury. See Figure 2.

10.8.2.4. Any gauge that fails calibration is to be tagged out and sent for repair.

11. CALCULATIONS / DATA REDUCTION

11.1. Not Applicable.

12. METHOD PERFORMANCE

12.1. Method performance is verified by analysis and certification of the screen can.

13. POLLUTION CONTROL

13.1. It is TestAmerica's policy to evaluate each procedure 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).

14. WASTE MANAGEMENT

14.1. 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 SANA-EHS-001.

14.2. Waste Streams Produced

14.2.1. Canisters that are no longer viable are purged with nitrogen, the valve removed, and the canister is either crushed with the laboratory trash or sent out for recycling.

14.2.2. Rough pumps generate Used Pump Oil, which is placed in one-liter polys and sent to the 90-day area for laboratory packing.

15. REFERENCES / CROSS-REFERENCES

15.1. EPA/625/R-96/010b, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, 2nd edition, January 1999

15.1.1. Compendium Method TO-14A, Determination of Volatile Organic Compounds (VOCs) in Ambient Air using Specially Prepared Canisters With Subsequent Analysis Gas Chromatographic Analysis

15.1.2. Compendium Method TO-15, Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)

15.2. TestAmerica Los Angeles QA Manual, current revision

15.3. TestAmerica Los Angeles SOP LA-MSA-014, Determination of Volatile Organics in Non-ambient Whole Air Samples using GC/MS-Scan Mode, Methods EPA TO-14A and EPA TO-15, current revision

15.4. TestAmerica Los Angeles SOP LA-MSA-015, Determination of Low-level Volatile Organics in Ambient / Indoor Whole Air Samples using GC/MS-Scan Mode, Methods EPA TO-14A and EPA TO-15, current revision

15.5. TestAmerica Los Angeles Safety SOP SANA-EHS-001, Sample and Chemical Waste Characterization, Collection, Storage and Disposal, current revision

15.6. TestAmerica Corporate QA SOP CW-Q-S-002, Writing an SOP, current revision

16. METHOD MODIFICATIONS

16.1. Not applicable.

17. ATTACHMENTS

17.1. Attachment 1: Canister QC Certification Sheet

17.2. Attachment 2: Flow Diagram of Automated Can Cleaning System

17.3. Attachment 3: Vacuum Gauge Calibration Worksheet

Attachment 1: Canister QC Certification Sheet

CANISTER QC
CERTIFICATION

TestAmerica

Certification Type: _____

Date Cleaned/Batch _____

Date of QC _____

Data File Number _____

CANISTER ID NUMBERS

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

The above canisters were cleaned as a batch. This certifies this batch contains no target analyte concentration greater than or equal to the method criteria for the "*Certification Type*" indicated above.

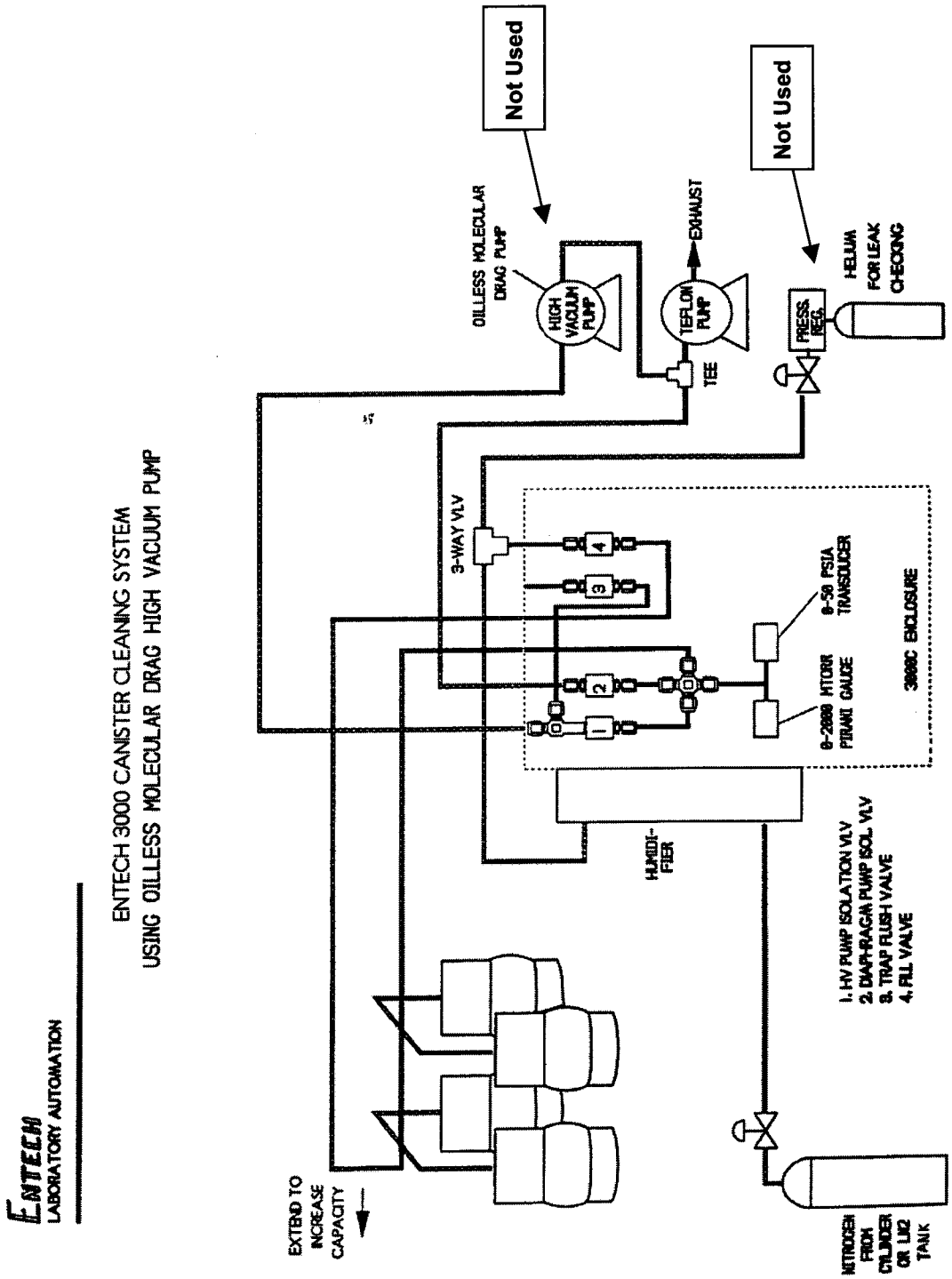
"*" INDICATES THE CAN OR CANS WHICH WERE SCREENED.

_____ Reviewed By:

_____ Date:

N:\CORDOCS\TestAmerica DOCs\Can QC Cert20070712.doc

Attachment 2: Flow Diagram of Automated Can Cleaning System



Attachment 3: Vacuum Gauge Calibration Worksheet

VACUUM GAUGE CALIBRATION - WORKSHEET

Client:	**Project:**
Calibrated Range: **Acceptable Range: ± 0.75 inch Hg**	**Date Calibrated:** **Calibrated By:**

DATE	GAUGE ID	READING Atmospheric pressure (0 ± 0.4psi)	READING Calibrated Gauge Vacuum	READING Gauge @ Vacuum (±0.75 in Hg. Calibrated Reading)	INTIALS	COMMENTS

18. REVISION HISTORY

18.1. This section has been added beginning with revision 7. Prior revisions are documented in QA files.

18.2. Changes to revision 6 implemented in revision 7:

18.2.1. The policy regarding the canister release schedule in section 1.4 was changed from seven days after final invoicing to seven days after mailing the final report.

18.2.2. The definition of a passivated canister was added in section 3. Hence, any collective reference to SUMMA canister and SILCO canister was changed to passivated canister.

18.2.3. The definition of a TO-can was added in section 3.

18.2.4. The term "process meters" was changed to "pressure gauges" in sections 10.1 and 10.1.2.

18.2.5. A statement that canisters may be released only after seven days from the date the final report was mailed was added in section 11.3. This section was also renumbered as 11.2.3 in the current revision.

18.2.6. The pressure reading identified in section 11.4.5 was changed from 0.20 psia to a range of 0.20 to 0.50 psia.

18.2.7. In section 11.4.13, a logbook title was changed from "Canisters and Regulators Logbook" to "Canisters and Regulators Cleaning Logbook."

18.2.8. The nitrogen pass-through duration identified in section 11.5.4.1 was changed from 30 and 45 minutes to 10-15 minutes.

18.2.9. References to method EPA TO-14 were changed to EPA TO-14A.

18.2.10. References to cold trap/liquid nitrogen from sections 6 and 7 were removed.

18.2.11. Other sections were modified for clerical corrections and formatting.

18.3. Changes to revision 7 implemented in revision 8:

18.3.1. This SOP has been formatted using the TestAmerica Corporate QA SOP template specified in SOP CW-Q-S-002.

18.3.2. All references to "Severn Trent Laboratories, Inc." or "STL" have been changed to "TestAmerica".

18.3.3. The canister's final evacuation vacuum specified in sections 2.2.5 and 11.3.8 was corrected from being 0.050 torr to being $\leq$ 0.050 torr. Note that section 11.3.8 has been renumbered as section 10.4.8 in revision 8.

18.3.4. The definitions for VFR, particulate filter, and pressure gauge have been added to section 3.

18.3.5. Section 5 (Safety) was revised in order to comply with the TestAmerica Corporate safety requirements.

18.3.6. Section 14 (Waste Management and Pollution Prevention) was divided into two sections (section 13 - Pollution Control and section 14 - Waste Management in revision 8), as specified in the TestAmerica Corporate QA SOP template. The specific discussions in each section were also revised according to the referenced template.

18.3.7. Additional SOP references were added to section 15. This section was also renamed "References / Cross-References", as specified in the TestAmerica Corporate QA SOP template.

18.3.8. Other sections were modified for clerical corrections and formatting.

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Los Angeles

SOP No.: LA-MSA-014, Rev. 8
Effective Date: 12/29/2006

Title: **DETERMINATION of VOLATILE ORGANICS in NON-AMBIENT WHOLE AIR SAMPLES using GC/MS-SCAN MODE**
[Methods EPA TO-14A and EPA TO-15]

Approvals (Signature/Date):

Dave Kammerer Air Lab Department Manager	9-17-7 Date	William Nash Environmental Health & Safety Coordinator	09/17/07 Date
Maria Friedman Quality Assurance Manager	9-17-2007 Date	Elizabeth Winger Laboratory Director	9/17/07 Date

This SOP was previously identified as SOP No. COI-MS-0003.

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17. SOP REVISION HISTORY 45

1. SCOPE AND APPLICATION

1.1. This standard operating procedure (SOP) is applicable to the analysis of volatile organic compounds (VOCs), having molecular weight in the general range of 40-200 g/mol and vapor pressure greater than 0.10 Torr at 25°C and 760 mm Hg in ambient air, by gas chromatography/mass spectroscopy (GC/MS) technique. This SOP is based on the EPA TO-14A/TO-15 method specifications and is applicable to various air matrices that include soil gas, landfill gas, and vapor treatment gases.

1.1.1. For low-level TO-14A/TO-15 analysis that is applicable to indoor and ambient air samples, refer to SOP COI-MS-0007.

1.2. Target compounds and their routine reporting limits (RLs) are provided in Table 1.

1.3. Additional compounds, which are not included in the standard list in Table 1, are identified as "add-ons" and can be found in Table 2.

2. SUMMARY OF METHOD

2.1. An air sample and internal standards (IS) are metered through a mass flow controller and concentrated onto a cryogenically cooled or adsorbent trap. After the internal standard and an appropriate amount of sample have been trapped, a valve is switched and the trap is heated to purge the trap's contents onto the GC column. The target compounds are analyzed with a mass selective detector operated in the scan mode.

3. DEFINITIONS

3.1. Batch – An analytical batch is defined as a set of up to 20 client samples of the same matrix processed using the same procedures and the same lot(s) of reagents within the same time period. A batch must contain a Laboratory Control Sample (LCS), LCS duplicate (LCSD), and a method blank (MB), but they do not count towards the maximum 20 samples in a batch.

3.1.1. The batch must be analyzed sequentially using the same instrument and instrument configuration within the same calibration event. That is, the same calibration curve, calibration factors, or response factors must be in effect throughout the analysis.

3.1.2. Field QC samples (e.g., trip blanks, equipment blanks, and field duplicates) count as individual samples; therefore, they add to the batch count. Sample reruns due to results outside the calibration range do not

add to the batch count as long as the re-analysis is within the same calibration event (as defined in Section 3.1.1).

3.2. Method Blank – The MB is a 6-L "screen can" (see SOP COI-QA-0005) humidified with 40 µL of deionized (DI) water, pressurized to 40 psia (pounds per square inch absolute) with ultra high purity (UHP) nitrogen (N_2), and then analyzed with each batch of samples. As stated in the referenced SOP, the "screen can" is often a sample canister that is being certified after cleaning. The MB is used to identify any background interference or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data. Internal standards and surrogates are added to the analytical trap and the MB is processed in the same manner as samples.

3.3. Laboratory Control Samples – LCSs are laboratory-generated samples used to monitor the laboratory's day-to-day performance. The LCS/LCSD is spiked with the target compounds in Table 1, from which a sublist may be reported, as defined by NELAC requirements (see sections 9.5.1 and 9.5.2). The LCS/LCSD is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS/LCSD results provides evidence that the laboratory is performing the method within accepted quality control (QC) guidelines for accuracy and precision. The LCS/LCSD is prepared from a source independent of the calibration standards. Other analytes may be required to meet project specific data quality objectives.

3.4. Surrogates – Surrogates are organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Although not required by the method, each sample, MB, LCS, and LCSD is spiked with surrogate standards. Surrogate spike recoveries may be evaluated against project-specific requirements by determining whether the concentration (measured as percent recovery) falls within the required limits. Surrogate compounds are listed in Table 1.

3.5. Passivated canister – Commonly referred to as SUMMA canister, SILCO canister, or TO-can in 1.0-liter, 1.8-liter, 6-liter, or 15-liter volumes.

3.6. SUMMA – A nickel electropolish passivation process in which the interior of a stainless steel sample container is de-activated and rendered inert to most VOCs. SUMMA canisters are not considered suitable for holding sulfur compounds.

3.7. TO-can – A spherical stainless steel container (which is the equivalent of a SUMMA canister) that is manufactured by Restek using a proprietary

electropolishing process and extensively cleaned using an ultrasonic method that ensures a high-quality, passivated surface that maintains the stability of TO-14A/TO-15 compounds during storage.

3.8. Tedlar – An inert plastic film used to manufacture sample bags for volatile organic compounds. Use of Tedlar bags as sample collection medium constitutes a modification to the method (see section 15.9).

3.9. Standard pressure is defined as 1.0 atmosphere, 14.6 psia (pounds per square inch absolute), 0 inch of mercury, and 0 psig, based on laboratory elevation and average barometric pressure.

Note: Full vacuum (0 psia) = 30 inches of mercury vacuum.

3.10. Standard molar volume is defined as 24.5 L/mol at room temperature of 25°C and standard pressure of 1 atmosphere.

4. INTERFERENCES

4.1. Gas regulators are cleaned by the manufacturer using Freon 113, which is one of the target compounds. Before use with either UHP N_2, hydrocarbon-free air, IS, or a target compound standard mix, each regulator should be purged a minimum of three times with the appropriate gas.

4.2. Contamination may occur in the sampling system if canisters are not properly cleaned prior to use. Canisters should not be used for the collection of samples until a batch blank analysis indicates that no target compounds are present above the RL, or a level previously agreed upon between STL and the client. Further information regarding the cleaning and certification of canisters may be found in SOP COI-QA-0005. All other sampling equipment including pumps, flow controllers, and filters must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

4.2.1. Canisters may also be individually certified clean as required by and at an additional cost to the client.

4.2.2. Canisters will be certified clean down to the method detection limits (MDL) of the target analytes of interest if sample results need to be evaluated down to those limits. However, the laboratory must be provided advanced notification of the requirement. Canister order must be placed with at least seven-day advanced notice or certification requirement may not be guaranteed.

4.3. Carryover may occur when samples with high levels of contaminants are analyzed. The sample immediately following a high-level sample should be re-analyzed if carryover is suspected.

4.4. Tedlar bags may contain low levels of target analytes.

4.5. Only compounds having both similar mass spectrum and GC retention time (RT) would be expected to interfere in the method. This situation most commonly occurs with structural isomers.

4.6. Matrix interferences may be caused by non-target contaminants that are present in the sample. The extent of matrix interference will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual (CSM), Lab Specific Addendum to the CSM, and this document.

5.2. Specific Safety Concerns and Requirements

5.2.1. Gas pressurized equipment is used in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment is over-pressurized.

5.2.2. The effluents from the sample splitters for the GC and the roughing pumps for the MS must be vented to a fume hood or at a minimum, must pass through a charcoal filter.

5.2.3. Both the GC and the MS contain elevated temperature zones. These zones must be cooled prior to an analyst or technician working on the unit

5.2.4. The MS is under deep vacuum and must be brought to atmospheric pressure before working on the source.

5.2.5. Due to high voltage risk, power to the GC and/or MS must be turned off or disconnected before work can be done on the instrument

5.2.6. Sampling canisters should never be pressurized over 40 psig.

5.2.7. Pressurized gas cylinders must be securely retained.

5.3. Primary Materials Used

5.3.1. 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	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
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.
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.
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. Vapors may cause irritation to the skin and eyes. Overexposure may cause lightheadedness, nausea, headache, and blurred vision.
Benzene	Flammable Poison Carcinogenic	1 ppm TWA	Toxic by ingestion, inhalation and absorption. Causes headache, nausea, dizziness, weakness and breathing difficulties. This material is irritating on contact with the skin and eyes and may cause permanent eye damage.

Material (1)	Hazards	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
Chloroform	Carcinogen Irritant	50 ppm Ceiling	Acts as a relatively potent anesthetic. Irritates respiratory tract and causes central nervous system effects, including headache, drowsiness, and dizziness. Causes skin irritation resulting in redness and pain and may be absorbed. Removes natural oils. Vapors cause pain and irritation to eyes. Splashes may cause severe irritation and possible eye damage.
Carbon Tetrachloride	Carcinogenic Poison	10ppm – TWA 200ppm STEL	Toxic by ingestion, inhalation and absorption. Causes headache, nausea, dizziness and narcosis. Contact with skin or eyes may cause irritation. Consumption of alcohol may increase toxic effects.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Gas chromatograph capable of sub-ambient temperature programming for the oven and with the jet separator option (Hewlett Packard 5890)

6.2. Mass-selective detector equipped with computer and appropriate software (Hewlett Packard 5970B with Chemstation data system and Target 4.0 Report Generation Software)

6.3. Canister autosampler with adsorbent trap (Tekmar AUTOCan)

6.4. Chromatographic-grade stainless steel or nickel tubing and stainless steel fittings

6.5. Chromatographic column DB-624 0.53 ID, or DB-VRX 0.45ID, 75 m length (J&W Scientific or equivalent)

6.6. Stainless steel vacuum/pressure gauge capable of measuring from 30 inches of mercury to 40 psig (Span Instruments or equivalent)

6.7. High precision vacuum gauge or process meter for making daily standards (Cole Parmer, Ashcroft or equivalent) - must be calibrated, at a minimum quarterly, against the master gauge

6.8. Pressure regulators for carrier gas and standards – 2-stage, stainless steel diaphragm (single stage acceptable for standards)

6.9. Passivated canisters (SUMMA or TO-can) – 1.0-L, 1.8-L, 6-L, 15-L (S.I.S., Restek, or equivalent)

6.10. Tedlar bag, 3-L or 1-L (SKC, ESS, or equivalent)

6.11. 7-micron filters (Nupro), or equivalent

6.12. Adjustable vacuum flow regulators (Valin or equivalent)

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. UHP N_2 and zero-grade air used for MB and preparing dilutions of samples and standards

7.1.2. UHP helium used as the gas chromatograph carrier gas

7.1.3. Liquid N_2

7.1.4. Purge-and-Trap grade water

7.2. Standards

7.2.1. Gas calibration stock standards, at a nominal concentration of 1 ppmv, containing the target compounds are purchased from commercial sources or are prepared from neat liquid in passivated canisters. Suppliers are required to provide certification of the analyte concentrations.

7.2.2. Add-on standards, either as neat liquid or as gaseous mix

7.2.3. Internal/surrogate stock standard mix at 250 ppbv. See Tables 1 and 10 for surrogates and internal standard compound list.

7.2.3.1. The surrogate mix is also used to tune the mass spectrometer.

7.2.4. Expiration dates for standards and reagents are based on vendor specification. If no vendor expiration date is assigned, the laboratory assigns an expiration date of two years from the date of receipt. Refer to

SOP SANA-QA-0007 for further information on standards and expiration dates. Expiration dates must be documented on the gas cylinders.

7.3. Standard Preparation

7.3.1. Static dilutions of the stock standard gas mixtures are made in 6- or 15-L passivated canisters to create working standards. A high precision vacuum gauge is flushed with UHP N_2 and attached to the top valve of a clean, evacuated canister, and the absolute pressure is recorded.

7.3.2. Depending on the concentration of each stock standard gas mixture, a particular pressure of each is added to the canister to achieve the desired concentration in the working standard.

7.3.3. Care should be taken to flush each regulator and transfer line with standard prior to transfer to the canister. After all of the stock standard mixes are added, the standard canister is pressurized with UHP N_2 to achieve the appropriate concentration.

7.3.4. Currently, the daily standard has a nominal concentration of 25 ppbv and is created by adding 1 psia each of the two 1 ppmv stock standard mixes and adding UHP N_2 to the canister to achieve a final pressure of 40 psia.

7.3.5. Detailed standard preparation steps may be found in logbooks NSL (Preparation of Gas Standards from Neat Liquids) and MSL (Preparation of Gas Standards from Gas Mixtures).

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Samples should be collected in certified clean (see section 4.2) passivated canisters. A 7-micron filter should be placed on the inlet of the canister to protect the valve from particulates. Canisters should never be pressurized over 40 psig.

8.2. The absolute pressure of the canister should be recorded before and after sample collection. See section 11.2 for sample preparation.

8.3. Samples should be protected from extreme temperatures.

8.4. Tedlar bag samples should be protected from sunlight.

8.5. Canister samples should be analyzed within **30 days from collection**.

8.5.1. Client or project-specific requirements may limit the holding time to 14 days.

8.6. Tedlar bag samples should be analyzed within **3 days from collection**.

8.6.1. Client or project-specific holding times may take precedence.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. Method Detection Limit – An MDL must be determined prior to the analysis of any sample. STL LA will generate a valid MDL for each analyte of interest. The MDL must be below the RL for each analyte. The procedure for the determination of the MDL is outlined in STL SOP S-Q-003 based on 40 CFR Part 136, Appendix B. MDL studies must be performed annually.

9.1.1.1. For add-on or non-standard analytes, an MDL study is required by NELAC. Additionally, a calibration curve must be generated before analyzing any samples, unless lesser requirements (e.g., a single-point calibration, which should be at the RL) are previously agreed to with the client. Any such agreed deviation from the method must be clearly documented in the report narrative.

9.1.1.2. A second-source verification standard may not be available for all add-on standards.

9.1.2. Demonstration of Capability (DOC) – Prior to the analysis of any client sample, four replicates of the laboratory control sample (LCS) containing all of the target analytes approximately at the mid range of the calibration curve must be analyzed and results compared to in-house quality control (QC) acceptance limits. These data are used to establish method performance in terms of accuracy and precision. Annual DOCs must be generated to ensure an analyst's continued proficiency in the method.

9.2. Control Limits

9.2.1. Control Limits – In-house historical control limits must be generated for surrogates and LCS/LCSD. These limits are updated annually. The

recovery limits are determined as the mean recovery ±3 standard deviations.

9.3. Surrogate Standards

9.3.1. Surrogates are not a method requirement. The laboratory routinely adds surrogates to all QC and samples and will report these results if only defined in a QAPP/SOW or at client's request.

9.3.2. Surrogate recoveries in samples, blanks and QC samples may be assessed to ensure that recoveries are within 70% - 130 % or within laboratory historical limits, if available. If any surrogates are outside limits and if surrogates are a project-specific requirement, the following corrective actions must be performed:

9.3.2.1. Check all calculations for error.

9.3.2.2. Ensure that instrument performance is acceptable.

9.3.2.3. Recalculate data and/or re-analyze if either of the above checks reveal a problem.

9.3.2.4. Re-analyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

9.3.3. It is only necessary to re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst has reason to believe that the repeated out of control results are due to problems other than matrix effect.

9.4. Method Blank

9.4.1. For each batch, an acceptable MB must be analyzed. The MB is analyzed after the calibration standards and LCS prior to sample analysis.

9.4.2. The MB must not contain any analyte of interest above the RL (except common laboratory contaminants, see below). An MB that does not meet this criterion must be re-analyzed unless no reportable concentrations of target analytes are present in the associated samples.

9.4.2.1. If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone), the data may be reported with qualifiers if the concentration of the analyte is less than five

times the RL. Such action should be done after consultation with the client.

9.4.2.2. Re-analysis of samples associated with an unacceptable MB is required when reportable concentrations are detected in the samples. If re-analysis is not possible due to limited sample volume or other constraints, the MB is reported and all associated samples are flagged and appropriate comments are stated in the report narrative.

9.4.2.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable MB, the data may be reported with qualifiers.

9.4.3. If surrogates are a project-specific requirement, then the MB must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the MB has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-analysis of the MB and affected samples will be required.

9.5. Laboratory Control Samples (LCS/LCSD)

9.5.1. For each batch of samples, an LCS/LCSD pair must be analyzed after the calibration standard and before the MB and samples. The LCS/LCSD is spiked with the target compounds in Table 1, from which a sublist may be reported, as defined by NELAC requirements (see below). Client-specific requirements may require additional compounds or even the full list of analytes.

9.5.2. NELAC Requirements on LCS/LCSD composition – The following criteria shall be used for determining the minimum number of analytes to be spiked. However, the laboratory shall ensure that all targeted components are included in the spike mixture over a two-year period (NELAC Appendix D, D.1.1.2.1c, June 5, 2003, pages 250 and 251 of 324):

9.5.2.1. For projects that include 1 – 10 targets, spike all components.

9.5.2.2. For projects that include 11 - 20 targets, spike at least 10 or 80%, whichever is greater.

9.5.2.3. For projects with more than 20 targets, spike at least 16 components.

9.5.3. NELAC Requirements on LCS/LCSD acceptance – The number of allowable exceedences is based on the number of analytes in the LCS/LCSD. If more analytes exceed the LCS/LCSD control limits than is allowed, or if any one analyte exceeds the marginal exceedence (ME) limits, the LCS/LCSD fails and corrective action is necessary (NELAC Appendix D, D.1.1.2.1e, June 5, 2003, pages 251 and 252 of 324):

9.5.3.1. ME is defined as being beyond the LCS/LCSD control limits but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean. Upper and lower ME limits are established to determine when corrective action is necessary. Use of the ME limits does not apply to target analyte lists with fewer than 11 analytes. Also, the ME must be random. If the same analyte exceeds the LCS/LCSD control limits repeatedly, it is an indication of a systematic problem that must be immediately corrected (see section 9.5.4).

9.5.3.2. The number of allowable ME is as follows:

9.5.3.2.1. >90 analytes in LCS/LCSD, 5 analytes allowed in ME of the LCS/LCSD control limits

9.5.3.2.2. 71 – 90 analytes in LCS/LCSD, 4 analytes allowed in ME of the LCS/LCSD control limits

9.5.3.2.3. 51 – 70 analytes in LCS/LCSD, 3 analytes allowed in ME of the LCS/LCSD control limits

9.5.3.2.4. 31 – 50 analytes in LCS/LCSD, 2 analytes allowed in ME of the LCS/LCSD control limits

9.5.3.2.5. 11 – 30 analytes in LCS/LCSD, 1 analyte allowed in ME of the LCS/LCSD control limits

9.5.3.2.6. <11 analytes in LCS/LCSD, no analyte analytes allowed in ME of the LCS/LCSD control limits

9.5.3.3. The ME limits are updated annually at the same time the LCS/LCSD limits are updated. A copy of the ME limits are distributed to the laboratory and is kept in the QA files.

9.5.4. Corrective Action for failed LCS/LCSD

9.5.4.1. If any analyte is outside established LCS/LCSD limits but within established ME limits, no further corrective action is necessary but a nonconformance memo (NCM) must be generated using the laboratory's electronic NCM system in order to determine the randomness of the exceedence, as required by NELAC (see section 9.5.3.1).

9.5.4.2. If any analyte is outside both the established LCS/LCSD limits and the established ME limits, both a corrective action and an NCM are required.

9.5.4.2.1. Evaluate the analytical run for errors and anomalies. Re-analyze the LCS.

9.5.4.2.2. Check the standard for appropriate pressure. Low pressure will often cause failure. Re-pressurize or prepare a new standard and re-analyze the LCS/LCSD.

9.5.4.2.3. Evaluate the instrument status and perform maintenance. Re-analyze the continuing calibration verification (CCV) standard and the LCS/LCSD, or recalibrate.

9.5.4.2.4. If the batch is not re-analyzed, the data must be flagged and the reasons for accepting the batch (e.g., insufficient sample volume for re-analysis) must be reported in an NCM and discussed in the report narrative.

9.5.4.2.5. If the analytes in the LCS exceed the upper control limits (of the LCS/LCSD and the ME) and no analytes are detected in any of the samples, they may be reported without qualification. Positive bias is not expected to impact ND results. However, an NCM is still required in order to

determine randomness of the exceedence, as required by NELAC (see section 9.5.3.1).

9.6. Internal standards in Samples

9.6.1. The IS areas are monitored for each shift by comparing the IS areas in each sample against those of the associated CCV standard.

9.6.2. Sample IS areas are considered acceptable if they fall between 60% to 140% (for TO-15 medium-level) or -50% to 200% (for TO-14A) of the CCV IS areas.

9.6.3. The RTs are considered acceptable if they fall within $\pm$20 seconds (0.33 minutes) of the IS RT of the associated CCV.

9.6.4. Any sample exceeding the above criteria must be re-analyzed. If the IS area fails upon re-analysis and the sample results are reported, the failure must be documented on the analysis benchsheet, an NCM created, and the failure discussed in the report narrative. Internal standards are listed in Table 10.

9.7. Sample Duplicate Analysis

9.7.1. A sample duplicate is analyzed and reported with every 20 samples, if requested.

9.7.2. The acceptance criterion for the duplicate analysis is an RPD $\leq$25 for target compounds that are >5X the RL. No criterion is established for duplicate results <5X the RL. The calculations are provided in section 12.3.4.

9.8. Nitrogen Check

9.8.1. Before a new N_2 cylinder is used for the pressurization of samples or standards, it must be analyzed as a blank and pass all the criteria in section 9.4.

9.9. Annual Gauge Calibration

9.9.1. The master gauge, used to calibrate the gauges in the laboratory for measuring cylinder and canister pressure or vacuum, must be certified annually. The certification process is performed by an outside calibrating agency.

9.10. Process Flow Meter Calibration

9.10.1. The process flow meter, used in the laboratory to set the flow regulators at the client-requested flow rates for time-weighted sampling events, must be certified quarterly. The certification process is performed by an outside calibrating agency.

10. CALIBRATION AND STANDARDIZATION

10.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked to establish that the system meets the standard mass spectral abundance criteria. See section 11.6.

10.2. A low-level static dilution of the stock standard gas mixtures is made in a 6- or 15-L passivated canister. The high precision vacuum gauge is flushed with UHP N_2 and attached to the top valve of the clean, evacuated canister. After recording the absolute pressure, 2.00 psia of each of the standard mixtures is added to the canister (each regulator and the transfer line should be flushed several times before transfer of standards to the canister). Close the canister valves and replace the high precision gauge with a vacuum/pressure gauge of known accuracy. Pressurize the canister with UHP N_2 to 40 psia. This will yield a standard with a nominal concentration of 50 ppbv, for most compounds, when using stock standards at 1 ppmv.

10.3. A high level static dilution of the stock standard gas mixtures is made in a 6- or 15-L passivated canister. The high precision vacuum gauge is flushed with UHP N_2 and attached to the top valve of the clean, evacuated canister. After recording the absolute pressure, 8.00 psia of each of the standard mixtures are added to the canister (each regulator and the transfer line should be flushed several times before transfer of standards to the canister). Close the canister valves and replace the high precision gauge with a vacuum/pressure gauge of known accuracy. Pressurize the canister with UHP N_2 to 40 psia. This will yield a standard with a nominal concentration of 200 ppbv.

10.4. Initial Calibration

10.4.1. An initial calibration (ICAL) curve consisting of a minimum of five points at different concentrations is analyzed to determine the linear working range of the analytical system for each compound. An average response factor (RF), or sometimes called the relative response factor

(RRF), and the percent relative standard deviation (%RSD) are calculated for each target analyte using the following equations:

10.4.1.1. Calculation for RRF:

$$RRF = \frac{Area\ cpd\ in\ Std.}{Area\ I.S.} X \frac{Conc.I.S.}{Conc.cpd\ in\ Std.}$$

The area of the primary quantitation ion is used in the calculation.

10.4.1.2. Calculation for %RSD:

$$\%RSD = \frac{Std.Dev.of\ RRFs}{Mean\ of\ RRFs} X 100$$

10.4.2. The ICAL is considered acceptable if 90% of the target analytes have a %RSD $\leq$ 30. If this criterion is not met, a new ICAL is prepared.

10.4.2.1. Client- or project-specific requirements may dictate that the laboratory adhere to the following TO-15 criteria: The %RSD for the RFs for <u>all</u> target analytes in the ICAL must be $\leq$30, with up to two target analytes that may have a %RSD of $\leq$40.

10.4.3. Linear or quadratic curve fits may be used with at least six calibration points. The correlation coefficient r (coefficient of determination for non-linear curves, r^2) must be $\geq$0.995; r^2 must be $\geq$0.990. Please refer to the following equations.

10.4.3.1. Calculation of Linear Fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where: C_{ex} = Concentration in extract, µg/mL
R_x = Response for analyte
R_{Is} = Response for internal standard
C_{is} = Concentration of internal standard
A = Intercept
B = Slope

10.4.3.2. Calculation of Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

Where: C_{ex} = Concentration in extract, µg/mL
R_x = Response for analyte
R_{Is} = Response for internal standard
C_{is} = Concentration of internal standard
A = Intercept
B = Slope
C = Curvature

10.4.3.3. In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. The analyst should consider instrument maintenance to improve the linearity of response.

10.4.4. The nominal concentrations of the standards are typically 2, 5, 10, 50, 100, and 500 ppbv but these may vary depending on the certified mix used to prepare the standards or the volume trapped. The low standard must be at or below the RL. The standards are run by varying the trapped volume of the two working standards from the "default" volume of 500 mL. For example, the 2, 5, and 50 ppbv standards are analyzed by trapping 20, 50, 500 mL, respectively, of a 50 ppbv working standard.

10.4.5. Internal Standards in the ICAL

10.4.5.1. The IS response at each calibration level must be 60% to 140% (for TO-15 medium-level) or –50% to 200% (for TO-14A) of the IS response in the mid-point calibration standard.

10.4.5.2. The RT shift for each of the IS at each calibration level must be within 20 seconds of the RT of the IS in the mid-point calibration standard.

10.4.5.3. Each analyte at each level must be within 0.06 relative retention time (RRT) units of the mean RRT.

10.4.5.4. Any calibration level exceeding the above criteria must be re-analyzed.

10.4.6. The analyst may elect to drop points from the calibration curve to improve subsequent quantitation. The following rules apply. However, for further guidance , see the current revision of STL Policy P-T-001, Selection of Calibration Points.

10.4.6.1. Points below the RL may be dropped as long as there is a point remaining at or below the RL.

10.4.6.2. High points may be dropped but at the expense of decreasing the linear range.

10.4.6.3. Calibration points in between the low and high ends may NOT be dropped.

10.4.7. Analyte quantitation must be performed off the initial calibration and not from daily continuing calibration standard analysis. Test results must be qualified in reports when analyte quantitation is based on the CCV at the client's request. This request must also be documented in the report narrative.

10.5. Initial Calibration Verification (second-source standard)

10.5.1. Each new ICAL must be verified using an initial calibration verification (ICV), or second-source standard, if available. A second-source standard may not be available for all add-on compounds. Additionally, due to the insufficient number of suppliers of second source standards, custom standards prepared from neat materials in the laboratory are not routinely verified.

10.5.2. Since the regulatory agencies have not provided guidance on second source verification, the following acceptance criteria are used: ± 30 percent difference (%D) for target analytes, with allowance for any four of the poor performers identified below to be at ±55 %D:

10.5.2.1. Acetone

10.5.2.2. Acetonitrile

10.5.2.3. Acrolein

10.5.2.4. Acrylonitrile

10.5.2.5. Benzyl chloride

10.5.2.6. 2-Butanone

10.5.2.7. 1,4-Dioxane

10.5.2.8. Hexachlorobutadiene

10.5.2.9. 2-Hexanone

10.5.2.10. 4-Methyl-2-pentanone

10.5.2.11. Methyl-t-butyl ether

10.5.2.12. Naphthalene

10.5.2.13. 2-Propanol

10.5.2.14. Propene

10.5.2.15. Tetrahydrofuran

10.5.2.16. 1,2,4-Trichlorobenzene

10.5.2.17. Vinyl acetate

10.5.3. The limits in section 10.5.2 are provided as guidance. If these criteria are not met, the following corrective actions must be performed:

10.5.3.1. Rerun the second source check standard.

10.5.3.2. Re-prepare or acquire a new standard.

10.5.3.3. Evaluate instrument conditions.

10.5.3.4. Regenerate a new ICAL.

10.5.4. The concentration of the ICV standards must be varied.

10.6. Continuing Calibration Verification (daily standard)

10.6.1. Unless the QC batch follows a new ICAL, for each QC batch or for every 24 hours of operation, a single-point continuing calibration verification (CCV) standard (50 ppbv) is analyzed to verify the ICAL average RF. The %D of the CCV RRF from the ICAL average RRF is calculated for each compound using the following equation:

10.6.1.1. Calculation for %D:

$$\%D = \frac{|\,Average RRF\ from\ IC - RRF\ CCV\,|}{Average RRF\ from\ IC} X100$$

10.6.2. The CCV is considered acceptable if 90% of the target analytes have a %D $\pm$30. If the above criterion is not met, a second CCV should be analyzed. If it continues to fail, the analytical system should be evaluated.

10.6.2.1. Common causes of failing standards are low pressure or contaminated standards, incorrect or leaking autosampler lines used, system contamination or carryover, and tune issues.

10.6.2.2. If any of the above conditions are found and corrected, this situation must be recorded on the injection log. Additionally, changes to instrument parameters or repairs should also be recorded in the instrument maintenance logbook.

10.6.2.3. After corrections and adjustments are made, another CCV may be analyzed.

11. PROCEDURE

11.1. One time procedural variation is allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using the laboratory's nonconformance reporting system and approved by a technical specialist and the Quality Assurance (QA) Manager. If contractually required, the client shall be notified. The NCM shall be filed in the project file.

11.2. Sample Preparation

11.2.1. The initial vacuum/pressure of the sample canister is checked by attaching a vacuum/pressure gauge to the canister. The gauge should be

rinsed before use with UHP N_2 by physically holding it against the gas outlet and flushing for 10 seconds. Canister vacuum/pressure is routinely increased to above ambient (24 6 psia) to facilitate screening and GC tests. The initial and final vacuum/pressure must be recorded in the sample pressurization logbook and on the individual canister field data sheet.

11.2.1.1. When the canister vacuum/pressure is increased, a dilution factor (DF) is calculated and is applied to results:

$$DF = \frac{Y_a}{X_a}$$

where: Xa = absolute canister pressure before dilution (initial pressure)
Ya = absolute canister pressure after dilution (final pressure)

11.2.1.2. The pressure DF must be compensated for by trapping more sample. For example, a sample received at 12.0 psia and pressurized to 24.6 psia has a DF of 2.05. If the sample is relatively clean, a "full strength", 1X dilution analysis would result in a trap volume of 1020 mL (2.05 X 500). The recorded volume is rounded up to three significant figures.

11.2.2. Canisters received where no sample was collected (i.e., trip blanks) are pressurized like screen blanks (see section 3.2), and are considered to have a DF =1.0.

11.2.3. Samples are screened to check for contamination before analysis or if suspected to contain significant contamination, using GC/FID analysis or other screening methods. Screening is performed to determine a proper dilution or the optimum volume of sample for the calibrated range, and to prevent overloading the analytical instrument. The screening instrument is generally calibrated at a single-point for common analytes of interest. Screening result printouts are called "screen reports". The following screening procedure is followed:

11.2.3.1. Remove the sample from Sample Control by completing the Internal Chain of Custody logbook.

11.2.3.2. Attach the sample to an active sample line on the screening instrument.

11.2.3.3. Start the screen sequence in the instrument data system.

11.2.3.4. Push START on the instrument.

11.2.3.5. Open sample container valve.

11.2.3.6. Collect data report and determine the dilution required for the analysis.

11.2.4. A sample that requires only a small dilution can be analyzed by trapping a volume less than the standard volume. The minimum volume that can be trapped is 10 mL. Larger dilutions will be prepared in a Tedlar bag.

11.2.4.1. Tedlar bag dilutions can also be used for reporting analytes that exceed calibration range in the original analysis.

11.2.4.2. Serial dilutions, as above, are documented in the run log.

11.3. Water Addition

11.3.1. The analyst should be aware that humidity plays an important role in the recovery of certain target compounds, particularly polar compounds, and should be prepared to add humidity to canisters where appropriate. The addition of water helps to stabilize the behavior of these compounds, which might otherwise interact with the interior surface of the canister or with the stainless-steel lines of the sample manifold.

11.3.2. Since it is not practical to know the relative humidity of all canisters received at the laboratory, the analyst should assume that canisters are received at approximately 80 percent relative humidity. When preparing canister dilutions, the analyst should attempt to preserve the relative humidity of canisters at a level that will minimize recovery loss due to low canister relative humidity.

11.3.3. Under normal laboratory conditions, a 6-L canister at ambient pressure will have a relative humidity of 100% if approximately 100 uL of water is in the canister.

11.3.3.1. The minimum relative humidity at which canisters containing polar analytes can be analyzed before polar target recovery is negatively affected is approximately 20 – 30%.

11.3.3.2. The minimum relative humidity at which canisters containing nonpolar analytes can be analyzed before nonpolar target recovery is negatively affected is approximately 10%.

11.4. Major Maintenance

11.4.1. A new initial calibration is necessary following major maintenance such as changing the column, cleaning or repairing the source, replacing filaments, changing electronics, replacing the multiplier or changing moisture or Tenax traps.

11.5. Minor Maintenance

11.5.1. Minor maintenance includes cleaning the injector port, replacing filters, changing the pump oil, autotuning, switching filaments, replacing the syringe or injector tower, change/refill the calibration vial, changing seals and o-rings, ballasting pump, replacing fuses, replacing roughing pumps or transfer lines.

11.6. Initial/Daily GC/MS Tuning

11.6.1. After a successful autotune as per manufacturer's recommendations, each instrument is manually tuned using perfluorotributylamine (PFTBA) so that the mass-to-charge ratio (m/z) 69 is 100%, m/z 131 is approximately 34%, and m/z 213 is approximately 36%. The width and axis parameters are set using the routines in the software. This initial tune should remain stable for extended periods of time, and retuning with PFTBA should not be necessary every day.

11.6.2. At the beginning of each 24-hour shift, prior to any analytical runs, the GC/MS system must be verified if acceptable tune performance criteria are achieved, by running a 4-bromofluorobenzene (BFB) tune standard. If any of the key ions fail the abundance criteria listed in Table 8 for TO-14A or Table 9 for TO-15, the system is considered out of tune and any subsequent sample/standard analysis is considered unacceptable.

11.6.3. For the AUTOCan tune verification, trap 28 mL of the IS canister containing BFB. Use the BFB.mtc method on the AUTOCan for a 30 mL/min flow rate.

11.6.4. Once the tuning run is complete (~ 6 minutes), evaluate BFB against Tables 8 or 9, as appropriate. If the criteria are not met, BFB is re-

analyzed and re-evaluated. If BFB continues to fail, the GC/MS system is evaluated.

11.6.4.1. Adjustments to the mass axis calibration, the electron multiplier voltage, or other tune parameters may be required. All parameter changes must be recorded in the instrument maintenance logbook.

11.7. Analysis

11.7.1. Analytical steps for the Tekmar AUTOCan with timing:

11.7.1.1. Special Bake (if necessary) – 5 min

11.7.1.2. Next Sample Pressure Check – 0.5 min

11.7.1.3. Trapping Volume – 10 mL to 1500 mL. For trapped volume over 100 mL, use flow rate of 100 mL/min. For trapped volume under 100 mL, use flow rate of 30mL/min. Use the TO14ASLO.mtc method on the AUTOCan for a 30 mL/min flow rate. Trap temperature is 10°C.

11.7.1.4. Dry Purge – 4 min. @ 35°C

Note: For a complete list of AUTOCan settings, see Table 4.

11.7.2. The daily CCV standard and the QC samples are analyzed in the same manner as client samples. After the CCV standard is analyzed and evaluated (section 10.6), the LCS/LCSD are analyzed and evaluated (section 9.5).

11.7.3. Before sample analysis, a method blank is analyzed and evaluated (see section 9.4).

11.7.4. Instrument Operation (Tekmar AUTOcan)

11.7.4.1. A sample or standard canister is attached to the AUTOCan at one of the 16 autosampler positions. Before the canister is opened, a "Leak Check" is run from the Teklink software. This assures that the valves and lines are leak-free for unattended analysis. The leak check also cleans the fittings and lines of possible carryover from previous samples. The

change in pressure for the leak checks (DeltaP) should be 0.7 psia or less.

11.7.4.2. Next, a schedule is constructed using the Teklink software. Schedules can be saved and recalled for routine analysis or custom built for sample runs where the trapping volumes may vary significantly. 100 mL of IS from position A is trapped on every analysis. After the IS is trapped, a sample or standard aliquot is trapped onto the trap with the IS. Trapping is followed by three minutes of dry purging. The cryo-focuser is then cooled to -175°C. The cryotrap is heated to 320°C and desorbs the analytes onto the cryo-focuser for three minutes. Finally, the cryo-focuser heats to 120°C and injects the sample onto the GC column. Trapping volume should be kept between 10 mL and 1500 mL (see section 11.7.1.3).

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Analyses

12.1.1. Two criteria must be satisfied to verify positive identification:

12.1.1.1. Elution of sample component at the same GC relative or absolute RT as those of the standard component

12.1.1.1.1. The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component.

12.1.1.1.2. As an option, RT must compare within 0.33 minutes of the standard component absolute RT. For reference, the RT standard must be run within the same 24-hour shift as the sample.

12.1.1.2. Correspondence or matching of the sample component and the standard component mass spectra

12.1.1.2.1. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

12.1.1.2.2. The relative intensities of ions specified in section 12.1.1.2.1 must agree within ±30% between the standard reference and sample spectra. For example, for an ion with an abundance of 50% in the reference spectra, the corresponding sample abundance must be between 20% and 80%. Standard reference mass spectra must be obtained on each individual GC/MS system.

12.1.1.3. If a compound cannot be verified by all of the criteria in the above sections but in the technical judgment of the analyst the identification is correct, then the compound may be reported with thorough documentation and discussed in the report narrative.

12.2. Quantitative Analysis

12.2.1. When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. Quantification will take place using the internal standard technique. A summary of primary and secondary ions for target compounds and internal standards may be found in Table 7.

12.2.2. A sample must be analyzed and reported at a dilution if one or more target compounds have an on-column amount above the upper calibration level. Dilutions are acceptable if at least one of the following criteria are met:

12.2.2.1. Any target analyte in the diluted sample exceeds 80 ppbv on column.

12.2.2.2. The peak height of any non-target analyte in the diluted sample exceeds the largest peak height of the highest calibration standard.

12.2.2.3. A heavy hydrocarbon matrix in the diluted sample raises the baseline two times that of the relative IS.

12.3. Tentatively Identified Compounds (TICs)

12.3.1. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analysis being conducted. The following sections identify the guidelines for making tentative identification:

12.3.1.1. Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.3.1.2. Relative intensities of the major ions should agree within ±30%.

12.3.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.3.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

12.3.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.3.1.6. Only peaks having a total ion current greater than 10% of the nearest eluting IS total ion current will be evaluated for reporting.

12.3.2. TICs will be given general names consisting of major functional groups and number of carbon atoms unless an RT reference is available.

12.3.3. When TICs are requested to be reported using specific compound names, the following procedure must be followed:

12.3.3.1. Choose characterized ions of the specific compounds from the mass spectrum.

12.3.3.2. Search ions from expected RT range or entire RT range if the RT of the specific compound is unknown or uncertain.

12.3.3.3. Add the requested compounds as unknown in Target Review mode.

12.3.4. Semi-quantitative results will be calculated for TICs using total ion current areas and assuming an RRF = 1.0.

12.3.5. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

12.4. All manual or re-integration of chromatograms must be documented in accordance with STL Policy S-Q-004. Documentation includes, as a minimum, before and after copies of the chromatograms with a reference to the reason for re-integration, dated, and initialed. All manual integrations must undergo a second-level review.

12.5. Calculations

12.5.1. The Target data system is set up to automatically quantitate the sample results based on a 500-mL sample size. The default reporting unit is ppbv (see section 13). If a different sample size was used and/or a canister sample was pressurized, the result must be adjusted as shown below:

$$Final\ result(ppbv) = raw\ result(ppbv) \times \frac{500ml}{sample\ volume\ injected} \times \frac{final\ psia}{initial\ psia}$$

12.5.2. Calculation for Determining Concentration of Compounds

$$Conc.Cpd(ppbv) = \frac{Area\ cpd\ in\ sample}{Area\ I.S.in\ sample} X \frac{Conc.I.S.}{average\ RRF\ from\ ICAL} X\ Dil.Factor\ \ (see\ 12.5.1)$$

Note: The area of the primary quantitation ion is used in the calculation.

12.5.3. Calculation for Percent Recovery (%Rec)

$$\%Rec = \frac{amount\ cpd.\ recovered}{amount\ cpd.\ spiked} \times 100\,\%$$

12.5.4. Calculation for Relative Percent Difference (RPD)

$$RPD = \frac{Value\ A - Value\ B}{Average\ of\ Values} X\,100$$

13. REPORTING

13.1. The standard reporting unit is ppbv (also ppb v/v). If results are to be reported in ng/L or ug/m^3, use the following equation:

$$result\ ppbv\ X \frac{Molecular\ weight\ of\ compound}{24.5} = results\ ng/L\ or\ ug/m^3$$

Note: 24.5 is the molar volume of ideal gas at 25°C and 1 atm.

13.2. Estimates of uncertainty are based upon LCS historical control limits.

13.3. "J" values (results below the RL but above the MDL) are reported on request only.

13.4. No conversion of analytical results to standard conditions is made.

14. WASTE MANAGEMENT AND POLLUTION PREVENTION

14.1. All waste will be disposed of in accordance with federal, state, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by and this procedure. This method is set up in accordance with section 13 of the CSM for "Waste Management and Pollution Prevention."

14.2. Waste Streams Produced

14.2.1. Expired standards in cylinders are returned to the manufacturer.

14.2.2. Tedlar bags are placed in sample bins in the warehouse and are disposed on a quarterly basis. Disposal involves the slashing of the bags in a hood and then placing the bag in the laboratory trash stream. Highly contaminated bags may need to be profiled according to the contaminant and "laboratory-packed".

15. REFERENCES

15.1. Method Source

15.1.1. "EPA Compendium Method TO-14A. Determination of Volatile Organic Compounds (VOCs) in Ambient Air using Specially Prepared Canisters With Subsequent Analysis Gas Chromatographic Analysis." January 1999.

15.1.2. "EPA Compendium Method TO-15. The Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)." January 1999.

15.2. STL SOP S-Q-003, Method Detection Limit Studies, current revision.

15.3. STL SOP S-Q-004, Acceptable Manual Integration Practices, current revision.

15.4. STL Policy P-T-001, Selection of Calibration Points, current revision.

15.5. STL LA LQM, current revision.

15.6. STL LA SOP SANA-QA-0007, Standards Preparation, Traceability, and Verification, current revision.

15.7. STL LA SOP COI-QA-0005, Releasing and Cleaning of Sample Canisters and Cleaning, Calibration, and Setting of Flow Regulators and Vacuum Gauges, current revision.

15.8. STL LA SOP COI-MS-0007, Determination of Low-level Volatile Organics (VOCs) in Ambient Air by GC/MS – Scan Mode Using EPA Methods TO-14A & TO-15, current revision.

15.9. National Environmental Laboratory Accreditation Conference (NELAC), EPA/600/R-04/003. June 2003.

15.10. Deviations from Methods

15.10.1. UHP N_2 is used for dilution/pressurization purposes.

15.10.2. Method TO-14A recommends the use of a 0.32-mm column coupled directly to the MSD. With the HP system, the MSD can only handle flow

of 1 mL/min or less. The 0.32-mm column provides ~ 3 mL/min. STL uses a 0.53-mm column through a jet separator.

15.10.3. Method TO-14A describes an inlet system that uses a vacuum to pull the sample through the trap. STL optionally uses the pressure of the sample canister to drive the sample through the trap.

15.10.4. Method TO-14A describes the use of a Nafion dryer to remove excess moisture from air matrices. STL does not use a Nafion dryer since polar compounds may be lost during this removal step. The AUTOCan uses an adsorbent trap with a dry purge step to control moisture.

15.10.5. Method TO-14A describes the BFB tune check to be a gas sample introduced via a sample loop. STL traps and analyzes BFB using the same analytical technique used with samples.

15.10.6. Method TO-14A was written for ambient air samples, however, this SOP was designed for use on "non-ambient" air samples that generally have high level of contamination or do not require sub ppbv RLs.

15.10.7. Methods TO-14A and TO-15 describe the use of passivated steel canisters for sampling and analysis. No mention is made of the use of Tedlar bags. STL LA analyzes samples in Tedlar bags for VOCs using the same procedures described herein. A modification to the method is noted in the narrative of the final report.

15.10.8. Method TO-15 describes a shelf life of thirty days for primary working standards. STL LA maintains these standards for longer periods of time according to the manufacturer's recommendation and the results of stability monitoring.

15.10.9. The methods indicate that in order for the ICAL to be acceptable, all compounds must have a %RSD <30 (with allowance for two that could be up to 40% in TO-15). For routine analysis, STL LA accepts the ICAL if 90% of the target compounds have a %RSD $\leq$30 and if the average of the %RSD for all target compounds is <30%. This modification accounts for analytical issues that arise for poor performing analytes.

15.10.10. For the continuing calibration criteria, Method TO-14A states that the RPD of each RF (in the CCV) from the mean RF of the ICAL curve should be <30%; for Method TO-15, the %D for each target compound must compare to the ICAL at $\pm$30%. For routine analysis, STL LA

accepts the CCV if 90% of the target compounds have a %D ±30. This modification accounts for analytical issues that arise for poor performing analytes.

15.10.11. Method TO-15 sets accuracy and precision criteria of ±30% and ≤25%, respectively. STL LA uses project-specific QC criteria or control limits based on historical data that may be wider than the method limits.

15.10.12. Surrogates are not required by the methods. This SOP adds surrogates to every sample to help monitor for matrix effects and method performance. However, surrogates are not reported unless requested.

15.10.13. Method TO-15 states that the scan time must give 10 scans per peak, not to exceed 1 second per scan. The GC/MS software is set for a sampling rate of 3, which corresponds to approximately 2 to 3 scans per second, depending on the instrument. See the GC/MS operator's manual or "help" on the software for more information about the sampling rate.

16. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

Table 1. Reporting, Accuracy, and Precision Limits for Target and Surrogate Compounds**

Compound	RL, ppbv	Control Limits %	RPD %
Acetone	10		
Benzene	2.0	70 - 130	30
Benzyl chloride	25		
Bromodichloromethane	2.0		
Bromoform	2.0		
Bromomethane	2.0		
2-Butanone (MEK)	10		
Carbon disulfide	10		
Carbon tetrachloride	2.0		
Chlorobenzene	2.0	70 - 130	30
Dibromochloromethane	2.0		
Chloroethane	4.0		
Chloroform	2.0	70 - 130	30
Chloromethane	4.0		
1,2-Dibromoethane (EDB)	2.0		

Compound	RL, ppbv	Control Limits %	RPD %
1,2-Dichlorobenzene	2.0	70 - 130	30
1,3-Dichlorobenzene	2.0		
1,4-Dichlorobenzene	2.0		
Dichlorodifluoromethane	2.0		
1,1-Dichloroethane	2.0	70 - 130	30
1,2-Dichloroethane	2.0		
cis-1,2-Dichloroethene	2.0		
trans-1,2-Dichloroethene	2.0		
1,1-Dichloroethene	2.0	65 - 120	30
1,2-Dichloropropane	2.0	65 - 130	30
cis-1,3-Dichloropropene	2.0		
trans-1,3-Dichloropropene	2.0		
1,2-Dichloro-1,1,2,2-tetrafluoroethane	2.0		
Ethylbenzene	2.0		
4-Ethyltoluene	2.0		
Hexachlorobutadiene	4.0		
2-Hexanone	10		
Methylene chloride	2.0	70 - 120	30
4-Methyl-2-pentanone (MIBK)	10		
Styrene	2.0		
1,1,2,2-Tetrachloroethane	2.0	65 - 130	30
Tetrachloroethene	2.0	70 - 130	30
Toluene*	2.0	75 - 125	30
1,2,4-Trichlorobenzene	5.0		
1,1,1-Trichloroethane	2.0	60 - 140	30
1,1,2-Trichloroethane	2.0		
Trichloroethene	2.0	70 - 125	30
Trichlorofluoromethane	2.0		
1,1,2-Trichloro-1,2,2-trifluoroethane	2.0		
1,2,4-Trimethylbenzene	3.0	65 - 130	30
1,3,5-Trimethylbenzene	3.0		
Vinyl acetate	10		
Vinyl chloride	2.0		

Compound	RL, ppbv	Control Limits %	RPD %
m-Xylene & p-Xylene	2.0	70 - 130	30
o-Xylene	2.0	70 - 130	30
Xylenes (total)	2.0		
4-Bromofluorobenzene (surrogate)	n/a	70 - 130	30
1,2-Dichloroethane-d4 (surrogate)	n/a	70 - 130	30
Toluene-d8 (surrogate)	n/a	70 - 130	30

*RL for Toluene in a Tedlar bag sample is 5.0 ppbv.
** The RLs and control limits listed are current values. Not all compounds are spiked in the LCS/LCSD. RLs and control limits are re-evaluated annually.

Table 2. Reporting Limits for Additional, Non-Standard Compounds

Compound	RL, ppbv
Acetonitrile	20
Acrolein	10
Acrylonitrile	10
alpha-Methylstyrene	2.0
Bromobenzene	10
1-Bromo-2-chloroethane	10
1,3-Butadiene	4.0
Butane	2.0
t-Butanol	10
n-Butylbenzene	2.0
Sec-Butylbenzene	2.0
tert-Butylbenzene	2.0
Chlorodifluoromethane	10
2-Chloroethyl vinyl ether	100
Allyl chloride	4.0
2-Chlorotoluene	2.0
4-Chlorotoluene	2.0
Cyclohexane	2.0
Cyclohexanone	10
1,2-Dibromo-3-chloropropane	10

Compound	RL, ppbv
Dibromomethane	2.0
trans-1,4-Dichloro-2-butene	40
1,1-Dichloro-1-fluoroethane	2.0
Dichlorofluoromethane	2.0
1,3-Dichloropropane	10
2,2-Dichloropropane	2.0
1,4-Dioxane	10
Ethanol	40
tert-Amyl methyl ether (TAME)	2.0
Ethyl-tert-butyl ether	2.0
Ethyl acetate	4.0
Diethyl ether	2.0
Ethyl methacrylate	5.0
n-Heptane	2.0
n-Hexane	2.0
Iodomethane	5.0
Isopropylbenzene	2.0
Diisopropyl ether (DIPE)	2.0
4-Isopropyltoluene (p-Cymene)	2.0
Methacrylonitrile	5.0
Methanol	25
Methylcyclohexane	10
Methyl methacrylate	2.0
Methyl tert-butyl ether (MTBE)	2.0
Methyl tert-butyl ether (MTBE)	10
Naphthalene	5.0
n-Nonane	10
n-Octane	10
Pentane	2.0
Propane	5.0
2-Propanol	10
n-Propylbenzene	2.0
Propylene	5.0
1,1,1,2-tetrachloroethane	2.0

Compound	RL, ppbv
Tetrahydrofuran	5.0
1,2,3-Trichlorobenzene	2.0
1,2,3-Trichloropropane	2.0
1,2,3-Trimethylbenzene	2.0
2,2,4-Trimethylpentane	2.0
Vinyl bromide	2.0
TPH (as Gasoline)	500
Total Non-Methane Hydrocarbons as Hexane	500

** The RLs listed are current values. RLs are re-evaluated based on annual MDL studies performed at the laboratory.

Table 3. Retention Time and Dynamic Range for Target Compounds

Compound	Approximate RT (min.)	Dynamic Range* (ppbv)
Dichlorodifluoromethane (Freon 12)	2.05	2-500
Chloromethane	2.62	4-500
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	2.66	2-500
Vinyl Chloride	2.89	2-500
Bromomethane	3.34	2-500
Chloroethane	3.52	4-500
Trichlorofluoromethane (11)	3.85	2-500
1,1-Dichloroethene	4.44	2-500
Carbon Disulfide	4.54	10-500
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	4.52	2-500
Acetone	4.62	10-500
Methylene Chloride	5.07	2-500
trans-1,2-Dichloroethene	5.38	2-500
1,1-Dichloroethane	5.91	2-500
Vinyl Acetate	6.13	10-500
cis-1,2-Dichloroethene	6.70	2-500
2-Butanone	6.84	10-500
Chloroform	7.23	2-500
1,1,1-Trichloroethane	7.33	2-500
Carbon Tetrachloride	7.56	2-500
Benzene	7.88	2-500
1,2-Dichloroethane	7.97	2-500
Trichloroethene	8.95	2-500

Compound	Approximate RT (min.)	Dynamic Range* (ppbv)
1,2-Dichloropropane	9.31	2-500
Bromodichloromethane	9.88	2-500
cis-1,3-Dichloropropene	10.70	2-500
4-Methyl-2-pentanone	11.11	10-500
Toluene	11.21	2-500
trans-1,3-Dichloropropene	11.82	2-500
1,1,2-Trichloroethane	12.12	2-500
Tetrachloroethene	12.19	2-500
2-Hexanone	12.78	30-500
Dibromochloromethane	12.80	2-500
1,2-Dibromoethane	12.90	2-500
Chlorobenzene	13.98	2-500
Ethylbenzene	14.33	2-500
1,4-and 1,3-(p,m)Xylene	14.61	2-1000
1,2-(ortho)Xylene	15.43	2-500
Styrene	15.49	2-500
Bromoform	15.78	2-500
1,1,2,2-Tetrachloroethane	17.18	2-500
Benzyl Chloride	17.30	10-500
4-Ethyltoluene	17.55	2-500
1,3,5-Trimethylbenzene	17.73	2-500
1,2,4-Trimethylbenzene	18.55	2-500
1,3-Dichlorobenzene	19.02	2-500
1,4-Dichlorobenzene	19.26	2-500
1,2-Dichlorobenzene	19.83	2-500
1,2,4-Trichlorobenzene	21.32	20-500
Hexachlorobutadiene	21.52	4-500

*Ranges may change based on the initial calibration results achieved.

Table 4. Tekmar AUTOcan Operating Conditions

Method file: TO14A.mtc	**METHOD FILE LIST**
GC Start Option	End of Desorb
GC Cycle Time	3 Minutes
Cryo	On
Line Temp	120°C
Valve Temp	120°C
MCS Line Temp*	40°C
Trap Standby Temp	50°C
Cryo Standby Temp	70°C
MFC Standby Flow	60 mL/min
Trap Cool Temp	10°C
MFC Transfer Flow	100 mL/min**
Dry Purge Time	4 Minutes
Dry Purge Temp	35°C
Dry Purge Flow	100 mL/min
Desorb Preheat Temp	50°C
Trap Desorb Time	3 Minutes
Trap Desorb Temp	320°C
Cryo Cool Temp	-175°C
Cryo Inject Time	1 Minute
Cryo Inject Temp	120°C
Trap Bake Time	10 Minutes
Trap Bake Temp	335°C
MCS Bake Temp*	40°C
MCS Cool*	40°C

*MCS is not used for this application.
**For trapping volume < 100 mL, a separate method (TO14SLO.mtc) is used with a 30 mL/min transfer flow.

TABLE 5. BFB GC Operating Conditions

Method file: BFB.M (for HP5890 with AUTOCan autosampler)				
METHOD FILE LIST				
Method file:	**BFB.M**	GC Type: 5890 Column: Cap	Run type: SCAN,GC,E1 Splitless: Yes	
Temperature (°C):	**Inj.P**	**Intfc**	**Source**	
	N/A	200	280	
GC/DIP		**LEVEL A**	**LEVEL B**	**POST RUN**
Temp 1	180.0*	0.0	0.0	0.0
Time 1	4.40	0.0	0.0	0.0
Rate	0.0	0.0	0.0	
Temp 2	0.0	0.0	0.0	
Time	0.0	0.0	0.0	
Oven equilibration Time.	0.00 min			
Run time:	4.40			
Scan Start time	3.80			
Scan Parameters:	Mass Range:	28.5 to 270		
	Multiplier voltage:	Varies	Number of A/D samples:	8
	Threshold:	175 counts		

*Isothermal at 180°C.

TABLE 6. GC Analytical Method

Method file: TO14A.M (for HP5890 with AUTOCan autosampler)				
METHOD FILE LIST				
Method file:	**TO14A.M**	GC Type: 5890	Run type: SCAN,GC,E1	
		Column: Cap	Splitless: Yes	
Temperature (°C):	**Inj.P**	**Intfc**	**Source**	
	N/A	200	280	
GC/DIP		**LEVEL A**	**LEVEL B**	**POST RUN**
Temp 1	40	170	0.0	0.0
Time 1	4.0	0.0	0.0	0.0
Rate	8	40	0.0	
Temp 2	170	230.0	0.0	
Time	0.0	0.0	0.0	
Oven equilibration Time.	0.00 min			
Run time:	Approx. 23.75			
Scan Start time	Approx. 0.10			
Scan Parameters:	Mass Range:	28.5 to 270		
	Multiplier voltage:	Varies	Number of A/D samples:	8
	Threshold:	175 counts		

TABLE 7. VOC Key Ions

Constituent	Primary Ion	Secondary Ions		
Bromochloromethane I.S. #1	49	130	128	
Dichlorodifluoromethane (Freon 12)	85	87	50	
Chloromethane	52	***50**		
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	135	85	87	
Vinyl Chloride	62	64		
Bromomethane	94	96	79	
Chloroethane	64	66	49	
Trichlorofluoromethane (11)	101	103	66	
1,1-Dichloroethene	61	96	63	98
Carbon Disulfide	76	78	44	
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	101	***151**	103	85
Acetone	43	***58**		
Methylene Chloride	49	84	86	

Constituent	Primary Ion	Secondary Ions		
trans-1,2-Dichloroethene	61	96	98	63
1,1-Dichloroethane	63	65	83	
Vinyl Acetate	43	44	86	42
cis-1,2-Dichloroethene	61	96	98	63
2-Butanone	72	57	43	
Chloroform	83	85	47	
1,1,1-Trichloroethane	97	99	61	
Carbon Tetrachloride	117	119	121	82
1,4-Difluorobenzene I.S. #2	114	63	88	
Benzene	78	50	52	77
1,2-Dichloroethane	62	64	49	98
Trichloroethene	130	95	132	97
1,2-Dichloropropane	63	62	41	39
Bromodichloromethane	83	85	129	
cis-1,3-Dichloropropene	75	77	39	
4-Methyl-2-pentanone	43	58	85	100
Toluene	91	65	92	
Chlorobenzene-d5 I.S. #3	117	52	54	82
trans-1,3-Dichloropropene	75	77	39	
1,1,2-Trichloroethane	97	83	85	61
Tetrachloroethene	166	129	131	164
2-Hexanone	58	43	57	100
Dibromochloromethane	129	127	208	131
1,2-Dibromoethane	107	109	188	
Chlorobenzene	112	77	114	
Ethylbenzene	91	106	65	51
1,4-and 1,3-(p,m)Xylene	91	***106**	105	77
1,2-(ortho)Xylene	91	***106**	105	77
Styrene	104	***78**	103	51
Bromoform	173	171	175	93
1,1,2,2-Tetrachloroethane	83	85	133	131
Benzyl Chloride	91	126	63	
4-Ethyltoluene	105	120	77	
1,3,5-Trimethylbenzene	105	120	77	
1,2,4-Trimethylbenzene	105	120	77	
1,3-Dichlorobenzene	146	148	111	75
1,4-Dichlorobenzene	146	148	111	75

Constituent	Primary Ion	Secondary Ions		
1,2-Dichlorobenzene	146	148	111	75
1,2,4-Trichlorobenzene	180	182	109	145
Hexachlorobutadiene	225	227	223	
1,2-Dicholoroethane-d4 (S)	65	67		
Toluene-d8 (S)	98	100		
4-Bromofluorobenzene (S)	95	174	176	

*** Primary Ions for DB-VRX column due to a different elution order.**

Table 8. TO14A BFB Criteria

Mass	*Ion Abundance Criteria*
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2.0% of mass 174
174	>50% of mass 95
175	5.0 to 9.0% of mass 174
176	>95% but <101% of mass 174
177	5.0 to 9.0% of mass 176

Table 9. TO15 BFB Criteria

Mass	*Ion Abundance Criteria*
50	8.0 to 40% of mass 95
75	30 to 66% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2% of mass 174
174	50 to 120% of mass 95
175	4.0 to 9.0% of mass 174
176	93% to 101% of mass 174
177	5.0 to 9.0% of mass 176

Table 10. Internal Standards

Bromochloromethane
1,4-Difluorobenzene
Chlorobenzene-d5

17. SOP REVISION HISTORY

17.1. This section has been added beginning with revision 8. Prior revisions are documented in the QA files.

17.2. Changes to revision 7 implemented in revision 8:

17.2.1. The SOP title was corrected to indicate that the SOP is specifically used for the analysis of volatiles in non-ambient whole air samples by either method TO-14A or TO-15. This discussion was also added as a method deviation in section 15.10.6. Sole reference to TO-14A in some sections was also replaced with reference to both methods TO-14A and TO-15, as appropriate.

17.2.2. Specific references to "SUMMA canister" have been modified to either "passivated canister" or "canister". The latest revisions of Methods TO-14A and TO-15 have generalized the reference to "SUMMA canister" to other specially prepared canisters.

17.2.3. Sections 3 was expanded to include additional definition of terms used in this SOP.

17.2.4. The acceptance criterion for a "screen canister" discussed in section 4.2 was corrected, from having no target compounds above 0.20 ppbv to having no target compounds above the RL, in order to reflect the laboratory's current practice. Additionally, sections 4.2.1 and 4.2.2 were added to define additional canister certification requirements that may be requested from the laboratory.

17.2.5. The interference from high levels of carbon dioxide/moisture in the samples have been addressed by the laboratory and hence, no longer discussed in the SOP. This discussion used to be in section 4.3 of SOP revision 7.

17.2.6. Other sources of interferences that may possibly affect method performance were added in section 4.

17.2.7. Section 5 (Safety) was modified in order to comply with the requirements of the CSM.

17.2.8. Section 7 was expanded to identify reagents from standards. Additionally, a standards preparation section was added. Purge-and-trap grade water was added as a reagent (used for humidifying samples).

17.2.9. The NELAC requirements for LCS reporting were added as sections 9.5.1, 9.5.2, and 9.5.3 in the SOP's current revision.

17.2.10. Enhanced corrective action measures, when the LCS and/or LCSD fail, were also added as section 9.5.4 in the SOP's current revision.

17.2.11. The discussion in section 10.8 regarding IS evaluation in samples was moved to section 9.6 in the SOP's current revision.

17.2.12. Section 9.7 was added to address the acceptance criteria for sample duplicate analysis, if requested by client.

17.2.13. Section 9.8 was added to address the contamination check on the N_2 supply used to pressurize samples.

17.2.14. Section 9.9 was added to address the annual certification required for the master gauge that is used to calibrate the gauges used for samples and standards.

17.2.15. Section 9.10 was added to address the quarterly certification required for the process flow meter, which is used to set-up the flow rates of the flow regulators used by clients, for time-weighted sampling events.

17.2.16. The amount of standard mixtures used in preparing the low-level and the high-level dilution of the stock standard was corrected, in sections 10.2 and 10.3, respectively, in order to reflect the laboratory's current procedure.

17.2.17. The ICAL acceptance criteria in section 10.4.2 and subsection were corrected, in order to reflect the laboratory's current practice.

17.2.18. The method TO-15 ICAL acceptance criteria was added as section 10.4.2.1, to serve as reference when client-specific requirements dictate its use.

17.2.19. Use of linear or quadratic curve fits was addressed and added as section 10.4.3.

17.2.20. The discussion regarding IS evaluation in the ICAL was added as section 10.4.5 in the SOP's current revision.

17.2.21. Section 10.4.6 was added to address the criteria to be used when it becomes necessary to drop points from the ICAL.

17.2.22. Section 10.4.7 was added to address the use of the ICAL RF when calculating results and what to do if client-specific requirements dictate otherwise.

17.2.23. Section 10.5 was modified to address the new acceptance criteria for the ICV (second source) standard.

17.2.24. The CCV acceptance criteria in section 10.6 and subsections were corrected, in order to reflect the laboratory's current practice. Corrective actions to be performed when CCV fails were also addressed.

17.2.25. The number of seconds required to flush the pressurization gas line prior to each sample preparation/pressurization was defined in section 11.2.1.

17.2.26. The dilution factor used in the analysis of trip blanks was defined in section 11.2.2.

17.2.27. The discussion in section 11.2.3 regarding sample screening was expanded.

17.2.28. Section 11.2.4 was added to address the minimum volume of sample that can be trapped in the GC/MS system and the possible resort to Tedlar bag dilution when original analysis exceeds calibration range.

17.2.29. The guidelines for sample humidification, when deemed necessary, was addressed and may be found as section 11.3 in the SOP's current revision.

17.2.30. Section 11.4 was added to address the requirement for a new calibration curve, after major changes to the GC/MS system occurred.

17.2.31. Section 11.5 was added to provide some examples of minor maintenance to the GC/MS system.

17.2.32. Corrective actions (and their proper documentation) when BFB tunes fail were addressed and added as section 11.6.4.1.

17.2.33. The trapping volume range (as indicated in section 12.1) used in the Tekmar AUTOcan system was corrected from 50-2000 mL to 10-1500 mL. This discussion may now be found in section 11.7.1.3 of the SOP's current revision.

17.2.34. The operation procedure for the Tekmar AUTOcan discussed in section 12.4 was rewritten to reflect the laboratory's current practice.

17.2.35. The RT acceptance criterion discussed in section 13.1.1.2 was corrected from 0.5 minutes to 0.33 minutes, in order to reflect the laboratory's current practice. This discussion may now be found in section 12.1.1.1.2 of the SOP's current revision.

17.2.36. The relative ion intensities acceptance criterion discussed in sections 13.1.1.3 and 13.1.2.1 was corrected from $\pm$20% to $\pm$30%, in order to reflect the laboratory's current practice. This discussion may now be found in sections 12.1.1.2.2 and 12.3.1.2 of the SOP's current revision.

17.2.37. The proper documentation procedure to be followed by the laboratory when manual peak integration is performed was added and may be found as section 12.4 of the SOP's current revision.

17.2.38. References to the use of the NELAC document for QA guidance and the use of certain corporate policies and SOPs was added in section 17. The reference section may now be found as section 15 in the SOP's current revision.

17.2.39. Table 1 was updated to reflect the laboratory's current RLs and LCS/LCSD and surrogate acceptance limits.

17.2.40. Table 2 was updated to reflect the laboratory's current add-on list and their corresponding RLs.

17.2.41. Tables 8, 9, and 10 were added in order to supply information regarding BFB acceptance criteria for methods TO-14A and TO-15, and the internal standards used in the analysis.

17.2.42. All other sections were modified only for clerical corrections.

STL

STL Los Angeles

FACILITY SOP ATTACHMENT

SOP NUMBER: COI-MS-0003 rev 8	**CHANGE FORM ID:** CF1

SOP TITLE: Determination of Volatile Organics in Non-Ambient Whole Air Samples by GC/MS-Scan Mode using EPA Methods TO-14A & TO-15

REASON FOR ADDITION OR CHANGE: To change the corrective action for failed CCVs to comply with NELAC requirements.

CHANGE OR ADDITION:

Change section 10.6.2 to the following:

10.6.2. The CCV is considered acceptable if 90% of the target analytes have a %D ±30. If the CCV fails acceptance criteria, corrective actions must be performed. Per NELAC requirements, if routine corrective action procedures fail to produce a second consecutive (immediate) CCV within acceptance criterion, then either the laboratory has to demonstrate acceptable performance after corrective action with two consecutive CCVs, or a new ICAL must be generated.

Change section 10.6.2.3 to the following:

10.6.2.3. After maintenance or repair, the CCV (if this still meets the NELAC requirement stated in section 10.6.2) may be re-analyzed. Otherwise, a new ICAL is required.

Prepared By: William Daystrom

***APPROVED BY:**

Technical Review Signature	Date 2-16-7
Quality Assurance Manager	Date 2-16-2007
Environmental Health and Safety Coordinator	Date 02/16/2007
Laboratory Manager	Date 2/16/07

*Should be the same signature authorities of SOP being revised.

SEVERN TRENT STL

STL Los Angeles

FACILITY SOP ATTACHMENT

SOP NUMBER: COI-MS-0003 rev. 8	**CHANGE FORM ID:** CF2

SOP TITLE: Determination of Volatile Organics in Non-Ambient Whole Air Samples by GC/MS-Scan Mode using EPA Methods TO-14A & TO-15

REASON FOR ADDITION OR CHANGE: To add the calibration and analysis procedures for reporting multi-component analytes, TPH as Gasoline or TNMOC as Hexane.

CHANGE OR ADDITION:

Add the following subsections to section 10:

10.7. Multi-component analytes are reported from the total ion chromatogram as Total Petroleum Hydrocarbons (TPH) as Gasoline or Total Non-Methane Organic Compounds (TNMOC) as Hexane.

10.7.1. For TNMOC as Hexane, a multi-point external standard calibration of at least five points is analyzed using the same acceptance criteria listed in section 10.4.2 or 10.4.3. The hexane calibration is taken from the standard multi-component ICAL using the hexane peak in the total ion chromatogram. After calibration, the peak is changed to an "area sum peak" which will calculate the analyte concentration from the sum of all peaks in the total ion chromatogram. If the internal standard and surrogates are included in the method as target analytes, the analytical data processing software will automatically remove their areas from the area summation.

10.7.2. For TPH as Gasoline, the gasoline calibration is performed using gasoline in nitrogen standards. Using varying volumes, two or three different concentrations can be used to generate the curve. The current calibration levels are 0.50, 5.0, 10, 25 and 50 ppmv. The areas of all the peaks approximately between 2.1 and 25 minutes are summed, excluding the internal standards and surrogate peaks.

10.7.3. The response factor for each calibration point is calculated using the following formula:

RF=Area of compound in standard/Concentration of standard

10.7.4. The ICAL acceptance criteria in section 10.4.2 and 10.4.3 are followed.

10.7.5. A CCV must be analyzed and compared to the criteria in section 10.6.2 each time samples requiring TPH as Gasoline or TNMOC as Hexane are analyzed.

10.7.6. A sample result is considered to exceed the calibration range when the peak height of the highest peak in the sample exceeds the peak height of the upper calibration level.

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FACILITY SOP ATTACHMENT

SOP NUMBER: COI-MS-0003 rev. 8	**CHANGE FORM ID:** CF2
Prepared By: Maria Friedman	
***APPROVED BY:**	
Technical Review Signature	Date 2-28-07
Quality Assurance Manager	Date 2-28-2007
Environmental Health and Safety Coordinator	Date 02/28/2007
Laboratory Manager	Date 2/28/07

*Should be the same signature authorities of SOP being revised.

EMLab P&K

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Back

Asbestos Analysis in Soils and Rock: CARB 435 and EPA Screening Protocol Modified (Qualitative and Semi-Quantitative) using PLM.

Environmental Microbiology Laboratory

Document Number: 100217	Origin Date: 11/8/05	Revision Number: 1.1	Revision Date: N/A

Introduction

1. The following procedure describes the protocol for CARB 435, 400 point counting technique and the EPA Screening Protocol Modified (Qualitative and Semi-Quantitative) using PLM for analyzing soils and rock.
2. The analyses will be performed in accordance with the California Air Resources Board (CARB) Method 435 for the determination of asbestos in serpentine aggregate samples, adopted on June 6, 1991.
3. This method is applicable to determining asbestos content of serpentine aggregate in storage piles, on conveyor belts, and on surfaces such as roads, shoulders and parking lots.
4. Sample preparation follows a standard CARB 435 prep method. If crushing of the sample is required, then the sample will be sent to an offsite location for being crushed. The entire sample will be dried at 135-150oC and then crushed to ~3/8" gravel size. If the submitted sample is >1 pint, the sample will be split using a 1/2" riffle splitter following ASTM Method C-702-98 to obtain a 1-pint aliquot. The entire 1-pint aliquot, or entire original sample, will then be pulverized to produce a nominal 200 mesh final product.
5. Small aliquots of the sample will be mounted on three separate microscope slides containing the appropriate refractive index oil and analyzed by PLM with EPA Method 600/R-93/116. If asbestos is identified and is detected to be less than 10% concentration by visual area estimate then an additional five sample mounts will be prepared and, the quantification of asbestos concentration will be obtained using the standard CARB Method 435 point count protocol.

References

1. EPA-600/M4-82-020 December 1982. Test Method; Interim method for the determination of asbestos in bulk insulation samples.

Definitions

1. PLM - Polarized Light Microscope

Materials and media

1. Polarizing light microscope capable of Kohler illumination and crossed polars
2. Red I compensator
3. 10X objective
4. Microscope slides
5. Coverglass
6. Refractive index liquids

7. Mortar and pestle
8. HEPA filter hood
9. Mechanical stage
10. Dispersion Staining Objective Lens: 10X
11. Eyepiece Reticule: 25 point Chalkey Point Array

Recommended Sampling Procedures

1. The clients will perform sampling and submit the samples to the laboratory for analysis. The following procedure is described to help in the communication with clients who might need some help in sampling protocols. Please note, while this procedure recommends common practices, other requirements might vary by state and/or agency.
2. The locations where grab samples will be taken are randomly chosen over the surface of the source. As for a storage pile, a minimum of three grab samples shall be taken even if the product pile contains less than 1000 tons of material.
3. If the location is conveyor belts, the grab samples shall be collected by stopping the belt a minimum of three times or using an automated sampler.
4. To collect sample from covered roads, a minimum of three samples shall be taken even if the road is less than one mile long. Grab samples shall not contain underlying soils.
5. For the covered areas like play yard or parking lot shall be characterized by taking grab samples from a minimum of three randomly chosen locations per acre. A minimum of three samples shall be taken even if the area is less than one acre. Grab samples shall not contain underlying soils.
6. To collect sample from covered road shoulder, the only difference is that a minimum of three grab samples shall be taken over a length of two miles of shoulder or over an area of two acres of shoulder surface. The word shoulder is meant to imply shoulders on both sides of the road.
7. Each of the grab samples shall be placed in the same appropriate sized sampling container.
8. This composite sample shall be crushed to produce a material with a nominal size of less than three-eights of an inch. Before crushing, the sample must be adequately dried. ASTM Method C-702-80 shall be used to reduce to size of the crushed grab sample to a 1-pint aliquot. The 1-pint aliquot, shall be further crushed using a Braun mill or equivalent to produce a material of which the majority shall be less than 200 Tyler mesh (around 0.75 microns).
9. An aliquot of the 200 mesh material shall be put into a labeled sealed container or Ziploc bag. The label must contain all the information listed:
    - Unique sample number
    - Facility name
    - Facility address or location where sample is taken
    - Date and time of sampling
    - Name of person performing sampling
10. The sample should then be sent to a lab for analysis. The analytical results are reported in percent asbestos fibers, which is the percent number of asbestos fibers contained in 400 randomly chosen particles of a bulk sample.
    Sample Preparation: The objective is to produce samples with a smooth (non-grainy) background in a medium with a refractive index of approximately 1.46. The technique below collapses the filter for easier focusing and produces permanent mounts, which are useful for quality control and inter laboratory comparison.

1. Quantification of Asbestos Content
    A. Visual and Point Count Methods
        I. Prepare three slides.
        II. View 10 fields per preparation. Identify all fibers.

        Exception I: If the sample is suspected of containing no asbestos a visual technique can be used to report that the sample does not contain asbestos. If all fibers are non-asbestos, report "No asbestos was detected by visual examination".

        Exception II: If the sample is suspected to have an asbestos content in excess of ten percent, a

visual technique can be used to report that the sample contains greater than ten percent asbestos. The standard operation procedure of the visual technique allowed in the National Institute of Standards and Technology's National Voluntary Laboratory Accreditation Program, Bulk Asbestos Handbook, National Institute of Standards and Technology publication number NISTIR 88-3879 dated October 1988.

III. If one fiber is determined to be asbestos, discontinue the visual method and perform the point counting technique as described below.

B. Point Counting Rules

I. Prepare 8 different preparations with the appropriate refractive index liquid of the representative sample. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. Count 50 non-empty points on each preparation using Chalkey point array to obtain a total occupied point count of 400 points.

1. Record the number of points positioned directly above each particle or fiber.
2. Record only one point if two points are positioned over same particle or fiber.
3. Record the number of points positioned on the edge of a particle or fiber.
4. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on the visual intersection, a point is scored for both categories.
5. If a test point lies over an ambiguous structure, no particle or fiber is recorded.
6. A fiber mat or bundle is counted as one fiber.
7. Asbestos fibers are defined as mineral fibers having an aspect ratio greater than 3:1.

Calculations

1. Calculate the asbestos percentage with the following equation:

% asbestos = a / t * 100

a = total number of asbestos points counted
t = total number of occupied points counted

For example if 400 occupied points were observed and 20 asbestos fibers were counted then:

% asbestos = 20 / 400 * 100
% asbestos = 5%

Safety

1. Inspect and wear appropriate protective clothing/equipment as procedure dictates and when necessary to avoid exposure.
2. Asbestos, a human carcinogen, should be handled only in an exhaust hood (equipped with a HEPA filter). Precautions should be taken when analyzing unknown samples, which may be asbestos, to preclude exposure to the person analyzing the sample and minimize the disruption to the parent material. Disposal of asbestos-containing materials should follow EPA Guidelines.
3. Do not remove any PACM from the contained cover/box at anytime when the sample is not within the bio-safety cabinet.
4. Assume all bulk samples received in the laboratory for analysis to have asbestos and so handle them accordingly.
5. Wash all areas of exposed skin prior to leaving the laboratory.
6. Always remain vigilant to any unsafe practices and conditions in the laboratory and immediately report such practices and/or conditions to the laboratory manager.
7. Periodic inspections will be made of the work area by the laboratory manager to ensure that the appropriate

PPE is being used at all times required.

Quality Control

1. The bulk materials received for asbestos analysis will be sealed using a Ziploc and stored in a bin in the designated storage room for a period of one month and then appropriately disposed off if no special request for holding has been made by the client.

Reporting

1. The asbestos and non-asbestos fiber concentration in the sample will be reported as a percentage.

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APPENDIX K-G

TESTAMERICA REFERENCE DATA SUMMARY

STL KNOXVILLE - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
11	Acetone	1.9	$\mu g/m^3$	0.47	$\mu g/m^3$	0	0	20	0	0	20
20	Acetonitrile	1.7	$\mu g/m^3$	0.67	$\mu g/m^3$	0	0	20	0	0	20
39	Acrolein	1.8	$\mu g/m^3$	0.57	$\mu g/m^3$	70	130	20	0	0	20
46	Acrylonitrile	11	$\mu g/m^3$	0.32	$\mu g/m^3$	0	0	0	0	0	0
2839	alpha-Methylstyrene	1.9	$\mu g/m^3$	0.72	$\mu g/m^3$	0	0	0	0	0	0
196	Benzene	0.64	$\mu g/m^3$	0.26	$\mu g/m^3$	70	130	30	0	0	20
220	Benzyl chloride	2.1	$\mu g/m^3$	0.52	$\mu g/m^3$	0	0	20	0	0	20
318	Bromobenzene	2.6	$\mu g/m^3$	1.3	$\mu g/m^3$	0	0	0	0	0	0
3357	1-Bromo-2-chloroethane	2.3	$\mu g/m^3$	1.2	$\mu g/m^3$	0	0	0	0	0	0
321	Bromochloromethane	1.6	$\mu g/m^3$	0.79	$\mu g/m^3$	0	0	0	0	0	0
323	Bromodichloromethane	1.3	$\mu g/m^3$	0.67	$\mu g/m^3$	0	0	20	0	0	20
333	Vinyl bromide	1.7	$\mu g/m^3$	0.87	$\mu g/m^3$	0	0	0	0	0	0
340	Bromoform	2.1	$\mu g/m^3$	1	$\mu g/m^3$	0	0	20	0	0	20
343	Bromomethane	0.78	$\mu g/m^3$	0.39	$\mu g/m^3$	0	0	20	0	0	20
355	1,3-Butadiene	1.8	$\mu g/m^3$	0.88	$\mu g/m^3$	0	0	0	0	0	0
3371	n-Butane	0.95	$\mu g/m^3$	0.47	$\mu g/m^3$	0	0	0	0	0	0
2728	t-Butanol	6	$\mu g/m^3$	2.4	$\mu g/m^3$	0	0	0	0	0	0
3271	2-Butanone (MEK)	2.9	$\mu g/m^3$	0.59	$\mu g/m^3$	0	0	20	0	0	20
1772	tert-Butyl alcohol	6	$\mu g/m^3$	2.4	$\mu g/m^3$	0	0	0	0	0	0
393	n-Butylbenzene	2.2	$\mu g/m^3$	0.82	$\mu g/m^3$	0	0	0	0	0	0
395	sec-Butylbenzene	2.2	$\mu g/m^3$	0.82	$\mu g/m^3$	0	0	0	0	0	0
398	tert-Butylbenzene	2.2	$\mu g/m^3$	0.82	$\mu g/m^3$	0	0	0	0	0	0
459	Carbon disulfide	3.1	$\mu g/m^3$	0.44	$\mu g/m^3$	0	0	20	0	0	20
463	Carbon tetrachloride	1.3	$\mu g/m^3$	0.44	$\mu g/m^3$	0	0	20	0	0	20
521	Chlorobenzene	0.92	$\mu g/m^3$	0.41	$\mu g/m^3$	70	130	30	0	0	20
535	Dibromochloromethane	1	$\mu g/m^3$	0.37	$\mu g/m^3$	0	0	20	0	0	20
550	Chloroethane	1	$\mu g/m^3$	0.37	$\mu g/m^3$	0	0	20	0	0	20
568	2-Chloroethyl vinyl ether	9.6	$\mu g/m^3$	4.8	$\mu g/m^3$	0	0	0	0	0	0
569	Chloroform	0.97	$\mu g/m^3$	0.29	$\mu g/m^3$	70	130	30	0	0	20
572	1-Chlorohexane	2.4	$\mu g/m^3$	1.1	$\mu g/m^3$	0	0	0	0	0	0
574	Chloromethane	0.82	$\mu g/m^3$	0.37	$\mu g/m^3$	0	0	20	0	0	20

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REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
606	Allyl chloride	1.2	µg/m^3	0.47	µg/m^3	0	0	0	0	0	0
614	2-Chlorotoluene	2.1	µg/m^3	0.78	µg/m^3	0	0	0	0	0	0
617	4-Chlorotoluene	2.1	µg/m^3	0.78	µg/m^3	0	0	0	0	0	0
669	Cyclohexane	1.4	µg/m^3	0.52	µg/m^3	0	0	0	0	0	0
539	1,2-Dibromo-3-chloropropane	3.9	µg/m^3	1.7	µg/m^3	0	0	0	0	0	0
3261	1,2-Dibromoethane (EDB)	1.5	µg/m^3	0.46	µg/m^3	0	0	20	0	0	20
888	Dibromomethane	2.8	µg/m^3	1.4	µg/m^3	0	0	0	0	0	0
904	1,2-Dichlorobenzene	1.2	µg/m^3	0.48	µg/m^3	70	130	30	0	0	20
907	1,3-Dichlorobenzene	1.2	µg/m^3	0.48	µg/m^3	0	0	20	0	0	20
910	1,4-Dichlorobenzene	1.2	µg/m^3	0.48	µg/m^3	0	0	20	0	0	20
922	trans-1,4-Dichloro-2-butene	2	µg/m^3	1	µg/m^3	0	0	0	0	0	0
924	Dichlorodifluoromethane	0.99	µg/m^3	0.3	µg/m^3	0	0	20	0	0	20
933	1,1-Dichloroethane	0.81	µg/m^3	0.2	µg/m^3	70	130	30	0	0	20
936	1,2-Dichloroethane	0.81	µg/m^3	0.4	µg/m^3	0	0	20	0	0	20
948	cis-1,2-Dichloroethene	0.79	µg/m^3	0.28	µg/m^3	0	0	20	0	0	20
950	trans-1,2-Dichloroethene	0.79	µg/m^3	0.28	µg/m^3	0	0	20	0	0	20
943	1,1-Dichloroethene	0.79	µg/m^3	0.24	µg/m^3	70	130	30	70	130	30
958	Dichlorofluoromethane	4.2	µg/m^3	0.63	µg/m^3	0	0	0	0	0	0
986	1,2-Dichloropropane	0.92	µg/m^3	0.37	µg/m^3	70	130	30	0	0	20
989	1,3-Dichloropropane	1.8	µg/m^3	0.69	µg/m^3	0	0	0	0	0	0
990	2,2-Dichloropropane	1.8	µg/m^3	0.91	µg/m^3	0	0	0	0	0	0
998	cis-1,3-Dichloropropene	0.91	µg/m^3	0.27	µg/m^3	0	0	20	0	0	20
1000	trans-1,3-Dichloropropene	0.91	µg/m^3	0.36	µg/m^3	0	0	20	0	0	20
996	1,1-Dichloropropene	1.8	µg/m^3	0.68	µg/m^3	0	0	0	0	0	0
1015	1,2-Dichloro-1,1,2,2-tetrafluoroethane	1.4	µg/m^3	0.63	µg/m^3	0	0	20	0	0	20
1585	Diisopropyl ether	4.2	µg/m^3	0.63	µg/m^3	0	0	0	0	0	0
1199	1,4-Dioxane	2	µg/m^3	0.4	µg/m^3	0	0	0	0	0	0
1290	Ethanol	3.8	µg/m^3	1.9	µg/m^3	0	0	0	0	0	0
5441	Tert-amyl methyl ether (TAME)	4.2	µg/m^3	0.63	µg/m^3	0	0	0	0	0	0
4370	Tert-amyl methyl ether	4.2	µg/m^3	0.63	µg/m^3	0	0	0	0	0	0
4372	Tert-butyl ethyl ether	4.2	µg/m^3	0.63	µg/m^3	0	0	0	0	0	0

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#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
5440	Ethyl-t-Butyl Ether (ETBE)	4.2	µg/m^3	0.63	µg/m^3	0	0	0	0	0	0
1325	Ethyl acetate	0.72	µg/m^3	0.29	µg/m^3	0	0	0	0	0	0
1332	Ethylbenzene	0.87	µg/m^3	0.3	µg/m^3	0	0	20	0	0	20
3790	4-Ethyltoluene	2	µg/m^3	0.34	µg/m^3	0	0	20	0	0	20
1017	Freon 114	1.4	µg/m^3	0.63	µg/m^3	0	0	0	0	0	0
1481	n-Heptane	4.1	µg/m^3	0.41	µg/m^3	0	0	0	0	0	0
1489	Hexachlorobutadiene	4.3	µg/m^3	2.1	µg/m^3	0	0	20	0	0	20
1514	n-Hexane	3.5	µg/m^3	0.35	µg/m^3	0	0	0	0	0	0
1515	2-Hexanone	1.6	µg/m^3	0.82	µg/m^3	0	0	20	0	0	20
1536	Iodomethane	2.3	µg/m^3	0.87	µg/m^3	0	0	0	0	0	0
1537	Methyl iodide	2.3	µg/m^3	0.87	µg/m^3	0	0	0	0	0	0
3442	Isobutane	0.95	µg/m^3	0.36	µg/m^3	0	0	0	0	0	0
1552	Isobutanol	6	µg/m^3	3	µg/m^3	0	0	0	0	0	0
4969	Cumene	2	µg/m^3	0.74	µg/m^3	0	0	0	0	0	0
1569	Isopropyl alcohol	1.2	µg/m^3	0.21	µg/m^3	0	0	0	0	0	0
1578	Isopropylbenzene	2	µg/m^3	0.74	µg/m^3	0	0	0	0	0	0
5439	Diisopropyl Ether (DIPE)	4.2	µg/m^3	0.63	µg/m^3	0	0	0	0	0	0
3795	4-Isopropyltoluene (p-Cymene)	2.2	µg/m^3	0.82	µg/m^3	0	0	0	0	0	0
1713	Methacrylonitrile	1.1	µg/m^3	0.41	µg/m^3	0	0	0	0	0	0
1811	Methylene chloride	0.69	µg/m^3	0.24	µg/m^3	65	125	30	65	125	30
1823	Methyl methacrylate	1.6	µg/m^3	0.61	µg/m^3	0	0	0	0	0	0
3283	4-Methyl-2-pentanone (MIBK)	1.6	µg/m^3	0.61	µg/m^3	0	0	20	0	0	20
3794	Methyl tert-butyl ether (MTBE)	3.6	µg/m^3	0.36	µg/m^3	0	0	20	0	0	20
1932	Naphthalene	2.6	µg/m^3	0.47	µg/m^3	0	0	0	0	0	0
2047	n-Octane	1.9	µg/m^3	0.7	µg/m^3	0	0	0	0	0	0
2125	Pentane	3	µg/m^3	0.44	µg/m^3	0	0	0	0	0	0
3440	Propane	1.8	µg/m^3	0.9	µg/m^3	0	0	0	0	0	0
1570	2-Propanol	1.2	µg/m^3	0.21	µg/m^3	0	0	0	0	0	0
2238	Propionitrile	1.1	µg/m^3	0.45	µg/m^3	0	0	0	0	0	0
2247	n-Propylbenzene	2	µg/m^3	0.74	µg/m^3	0	0	0	0	0	0
3448	Propylene	0.86	µg/m^3	0.34	µg/m^3	0	0	0	0	0	0

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SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
2355	Styrene	0.85	$\mu g/m^3$	0.26	$\mu g/m^3$	0	0	20	0	0	20
4968	1,1,1,2-Tetrachloroethane	2.7	$\mu g/m^3$	1.4	$\mu g/m^3$	0	0	0	0	0	0
2439	1,1,2,2-Tetrachloroethane	1.4	$\mu g/m^3$	0.62	$\mu g/m^3$	55	135	30	0	0	20
2445	Tetrachloroethene	1.4	$\mu g/m^3$	0.61	$\mu g/m^3$	70	130	30	0	0	20
2469	Tetrahydrofuran	5.9	$\mu g/m^3$	0.29	$\mu g/m^3$	0	0	0	0	0	0
2489	Toluene	1.1	$\mu g/m^3$	0.34	$\mu g/m^3$	65	135	30	0	0	20
2514	1,2,3-Trichlorobenzene	3	$\mu g/m^3$	0.74	$\mu g/m^3$	0	0	0	0	0	0
2515	1,2,4-Trichlorobenzene	5.9	$\mu g/m^3$	1.1	$\mu g/m^3$	0	0	20	0	0	20
2518	1,1,1-Trichloroethane	1.1	$\mu g/m^3$	0.54	$\mu g/m^3$	70	130	30	0	0	20
2522	1,1,2-Trichloroethane	1.1	$\mu g/m^3$	0.44	$\mu g/m^3$	0	0	20	0	0	20
2525	Trichloroethene	1.1	$\mu g/m^3$	0.43	$\mu g/m^3$	65	135	30	0	0	20
1428	Trichlorofluoromethane	2.2	$\mu g/m^3$	1.1	$\mu g/m^3$	0	0	20	0	0	20
2563	1,2,3-Trichloropropane	2.4	$\mu g/m^3$	1.2	$\mu g/m^3$	0	0	0	0	0	0
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	3.1	$\mu g/m^3$	1.2	$\mu g/m^3$	0	0	20	0	0	20
2764	Trichlorotrifluoroethane	3.1	$\mu g/m^3$	1.2	$\mu g/m^3$	0	0	0	0	0	0
2587	1,2,4-Trimethylbenzene	2	$\mu g/m^3$	0.39	$\mu g/m^3$	70	130	30	0	0	20
2592	1,3,5-Trimethylbenzene	2	$\mu g/m^3$	0.44	$\mu g/m^3$	0	0	20	0	0	20
1564	2,2,4-Trimethylpentane	2.3	$\mu g/m^3$	0.7	$\mu g/m^3$	0	0	0	0	0	0
2610	Vinyl acetate	3.5	$\mu g/m^3$	0.21	$\mu g/m^3$	0	0	20	0	0	20
2613	Vinyl chloride	0.51	$\mu g/m^3$	0.26	$\mu g/m^3$	0	0	20	0	0	20
2940	m-Xylene & p-Xylene	2.2	$\mu g/m^3$	0.87	$\mu g/m^3$	70	130	30	0	0	20
2623	o-Xylene	0.87	$\mu g/m^3$	0.26	$\mu g/m^3$	70	130	30	0	0	20
2627	Xylenes (total)	2.2	$\mu g/m^3$	0.26	$\mu g/m^3$	0	0	20	0	0	20
5635	1,1-Dichloro-1-fluoroethane	1.9	$\mu g/m^3$	0.95	$\mu g/m^3$	0	0	0	0	0	0
337	4-Bromofluorobenzene					70	130	0	0	0	0
2735	1,2-Dichloroethane-d4					70	130	0	0	0	0
2740	Toluene-d8					70	130	0	0	0	0

STL KNOXVILLE - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
456	Carbon dioxide	180	µg/L	31	µg/L	C	Y	18000	µg/L	80	120
2693	Carbon monoxide	11	µg/L	3.4	µg/L	C	Y			70	130
3377	Ethane	6.1	µg/L	1.5	µg/L	C	Y			70	130
3386	Ethene	5.7	µg/L	1.1	µg/L	C	Y			70	130
2841	Methane	1.3	µg/L	0.39	µg/L	C	Y	327	µg/L	80	120
2843	Nitrogen	11000	µg/L	7800	µg/L	C	Y			70	130
2842	Oxygen	2600	µg/L	400	µg/L	C	Y			70	130

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
11	Acetone	20	µg/kg	2.7	µg/kg	58	130	30	10	200	66
196	Benzene	5	µg/kg	0.23	µg/kg	75	129	20	55	138	20
323	Bromodichloromethane	5	µg/kg	0.48	µg/kg	72	125	30	47	131	51
340	Bromoform	5	µg/kg	0.65	µg/kg	43	149	30	26	141	64
343	Bromomethane	5	µg/kg	0.61	µg/kg	24	152	30	15	152	72
372	2-Butanone	20	µg/kg	1	µg/kg	27	200	46	21	195	60
459	Carbon disulfide	5	µg/kg	0.2	µg/kg	50	137	30	27	149	73
463	Carbon tetrachloride	5	µg/kg	0.45	µg/kg	57	137	30	32	143	68
521	Chlorobenzene	5	µg/kg	0.28	µg/kg	75	127	22	49	139	22
535	Dibromochloromethane	5	µg/kg	0.36	µg/kg	49	135	30	44	135	61
550	Chloroethane	5	µg/kg	0.54	µg/kg	31	144	30	32	140	66
569	Chloroform	5	µg/kg	0.4	µg/kg	73	115	30	59	128	46
574	Chloromethane	5	µg/kg	0.25	µg/kg	15	136	30	28	130	81
669	Cyclohexane	10	µg/kg	0.49	µg/kg	0	0	0	0	0	0
539	1,2-Dibromo-3-chloropropane	10	µg/kg	1.4	µg/kg	0	0	0	0	0	0
870	1,2-Dibromoethane	5	µg/kg	0.37	µg/kg	0	0	0	0	0	0
904	1,2-Dichlorobenzene	5	µg/kg	0.22	µg/kg	0	0	0	0	0	0
907	1,3-Dichlorobenzene	5	µg/kg	0.26	µg/kg	0	0	0	0	0	0
910	1,4-Dichlorobenzene	5	µg/kg	0.34	µg/kg	0	0	0	0	0	0
924	Dichlorodifluoromethane	5	µg/kg	0.4	µg/kg	0	0	0	0	0	0
933	1,1-Dichloroethane	5	µg/kg	0.32	µg/kg	77	119	30	56	130	54
936	1,2-Dichloroethane	5	µg/kg	0.48	µg/kg	78	121	30	56	126	38
948	cis-1,2-Dichloroethene	5	µg/kg	0.41	µg/kg	77	114	30	48	127	52
950	trans-1,2-Dichloroethene	5	µg/kg	0.55	µg/kg	68	117	30	47	127	58
943	1,1-Dichloroethene	5	µg/kg	0.6	µg/kg	55	142	27	43	147	27
986	1,2-Dichloropropane	5	µg/kg	0.35	µg/kg	78	116	30	54	125	43
998	cis-1,3-Dichloropropene	5	µg/kg	0.35	µg/kg	71	125	30	30	138	49
1000	trans-1,3-Dichloropropene	5	µg/kg	0.35	µg/kg	67	125	30	34	134	57
1332	Ethylbenzene	5	µg/kg	0.53	µg/kg	79	114	30	36	133	72
1515	2-Hexanone	20	µg/kg	0.84	µg/kg	29	200	41	20	190	70
1578	Isopropylbenzene	5	µg/kg	0.21	µg/kg	0	0	0	0	0	0
1774	Methyl acetate	10	µg/kg	0.77	µg/kg	0	0	0	0	0	0

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1799	Methylcyclohexane	10	µg/kg	0.46	µg/kg	0	0	0	0	0	0
1811	Methylene chloride	5	µg/kg	1.3	µg/kg	58	130	30	45	129	49
1845	4-Methyl-2-pentanone	20	µg/kg	0.54	µg/kg	68	142	60	42	143	60
2772	Methyl tert-butyl ether	20	µg/kg	0.28	µg/kg	70	130	30	70	130	30
2355	Styrene	5	µg/kg	0.2	µg/kg	80	114	30	23	136	65
2439	1,1,2,2-Tetrachloroethane	5	µg/kg	0.46	µg/kg	70	133	30	33	162	90
2445	Tetrachloroethene	5	µg/kg	0.83	µg/kg	72	120	30	31	137	81
2489	Toluene	5	µg/kg	0.29	µg/kg	71	130	24	46	147	24
2515	1,2,4-Trichlorobenzene	5	µg/kg	0.32	µg/kg	0	0	0	0	0	0
2518	1,1,1-Trichloroethane	5	µg/kg	0.76	µg/kg	67	123	30	48	132	57
2522	1,1,2-Trichloroethane	5	µg/kg	0.41	µg/kg	82	116	30	58	128	52
2525	Trichloroethene	5	µg/kg	0.41	µg/kg	70	131	23	46	143	23
1428	Trichlorofluoromethane	5	µg/kg	0.41	µg/kg	0	0	0	0	0	0
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	5	µg/kg	0.8	µg/kg	0	0	0	0	0	0
2613	Vinyl chloride	5	µg/kg	0.44	µg/kg	24	152	30	30	136	80
2627	Xylenes (total)	10	µg/kg	0.76	µg/kg	80	114	30	33	135	78
337	4-Bromofluorobenzene					47	158	0	47	158	0
2735	1,2-Dichloroethane-d4					61	130	0	61	130	0
2740	Toluene-d8					60	143	0	60	143	0
2863	Dibromofluoromethane					59	138	0	59	138	0
1	Acenaphthene	330	µg/kg	1.3	µg/kg	46	110	30	10	200	30
5	Acenaphthylene	330	µg/kg	1.2	µg/kg	47	110	30	10	200	30
24	Acetophenone	66.6	µg/kg	9	µg/kg	0	0	0	0	0	0
122	Anthracene	330	µg/kg	1.3	µg/kg	56	111	30	10	200	30
158	Atrazine	330	µg/kg	21	µg/kg	0	0	0	0	0	0
3398	Benzaldehyde	330	µg/kg	21	µg/kg	0	0	0	0	0	0
202	Benzo(a)anthracene	330	µg/kg	0.95	µg/kg	58	111	30	10	200	30
205	Benzo(b)fluoranthene	330	µg/kg	1.2	µg/kg	43	124	30	10	200	30
208	Benzo(k)fluoranthene	330	µg/kg	1.7	µg/kg	38	122	30	10	200	30
210	Benzo(ghi)perylene	330	µg/kg	1.3	µg/kg	44	120	30	10	200	30

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
211	Benzo(a)pyrene	330	µg/kg	1.3	µg/kg	44	115	30	10	200	30
3474	1,1'-Biphenyl	330	µg/kg	23	µg/kg	0	0	0	0	0	0
289	bis(2-Chloroethoxy)methane	330	µg/kg	22	µg/kg	42	110	30	36	110	30
293	bis(2-Chloroethyl) ether	330	µg/kg	2	µg/kg	41	110	30	32	118	30
302	bis(2-Ethylhexyl) phthalate	330	µg/kg	18	µg/kg	56	123	30	10	200	30
348	4-Bromophenyl phenyl ether	330	µg/kg	21	µg/kg	53	112	30	44	120	30
403	Butyl benzyl phthalate	330	µg/kg	19	µg/kg	57	121	30	43	138	30
5101	Caprolactam	330	µg/kg	37	µg/kg	0	0	0	0	0	0
2751	Carbazole	330	µg/kg	19	µg/kg	56	115	30	10	162	30
518	4-Chloroaniline	330	µg/kg	17	µg/kg	25	110	30	11	110	30
578	4-Chloro-3-methylphenol	330	µg/kg	21	µg/kg	42	110	30	32	117	30
589	2-Chloronaphthalene	330	µg/kg	22	µg/kg	46	110	30	40	110	30
600	2-Chlorophenol	330	µg/kg	26	µg/kg	39	110	30	32	110	30
602	4-Chlorophenyl phenyl ether	330	µg/kg	24	µg/kg	53	110	30	47	116	30
633	Chrysene	330	µg/kg	0.9	µg/kg	56	111	30	10	200	30
860	Dibenz(a,h)anthracene	330	µg/kg	1.3	µg/kg	45	122	30	10	200	30
863	Dibenzofuran	330	µg/kg	20	µg/kg	50	110	30	10	200	30
891	Di-n-butyl phthalate	330	µg/kg	19	µg/kg	57	119	30	31	145	30
918	3,3'-Dichlorobenzidine	1600	µg/kg	18	µg/kg	31	110	30	10	110	30
971	2,4-Dichlorophenol	330	µg/kg	20	µg/kg	40	110	30	33	110	30
1082	Diethyl phthalate	330	µg/kg	19	µg/kg	55	114	30	48	118	30
1145	2,4-Dimethylphenol	330	µg/kg	20	µg/kg	28	110	30	19	114	30
1149	Dimethyl phthalate	330	µg/kg	21	µg/kg	54	112	30	47	116	30
1167	4,6-Dinitro-2-methylphenol	1600	µg/kg	13	µg/kg	21	110	30	10	110	30
1187	2,4-Dinitrophenol	1600	µg/kg	83	µg/kg	10	110	30	10	110	30
1191	2,4-Dinitrotoluene	330	µg/kg	18	µg/kg	55	116	30	42	118	30
1193	2,6-Dinitrotoluene	330	µg/kg	21	µg/kg	54	115	30	28	137	30
1162	Di-n-octyl phthalate	330	µg/kg	18	µg/kg	45	123	30	10	182	30
1414	Fluoranthene	330	µg/kg	1.2	µg/kg	55	118	30	10	200	30
1417	Fluorene	330	µg/kg	1.2	µg/kg	51	110	30	10	187	30
1482	Hexachlorobenzene	330	µg/kg	2.1	µg/kg	51	110	30	37	122	30

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1489	Hexachlorobutadiene	330	µg/kg	26	µg/kg	39	110	30	30	110	30
1492	Hexachlorocyclopentadiene	1600	µg/kg	16	µg/kg	10	110	30	10	110	30
1497	Hexachloroethane	330	µg/kg	28	µg/kg	38	110	30	13	110	30
1535	Indeno(1,2,3-cd)pyrene	330	µg/kg	1.5	µg/kg	45	121	30	10	200	30
1566	Isophorone	330	µg/kg	21	µg/kg	46	117	30	32	129	30
1829	2-Methylnaphthalene	330	µg/kg	1.5	µg/kg	46	110	30	10	200	30
1851	2-Methylphenol	330	µg/kg	28	µg/kg	36	110	30	19	124	30
1857	4-Methylphenol	330	µg/kg	22	µg/kg	40	110	30	27	116	30
1932	Naphthalene	330	µg/kg	1.6	µg/kg	42	110	30	10	200	30
1960	2-Nitroaniline	1600	µg/kg	22	µg/kg	47	124	30	31	141	30
1964	3-Nitroaniline	1600	µg/kg	16	µg/kg	44	110	30	24	110	30
1968	4-Nitroaniline	1600	µg/kg	26	µg/kg	50	110	30	23	124	30
1972	Nitrobenzene	330	µg/kg	2.2	µg/kg	40	110	30	33	111	30
1998	2-Nitrophenol	330	µg/kg	19	µg/kg	35	110	30	17	110	30
2001	4-Nitrophenol	1600	µg/kg	110	µg/kg	24	117	30	10	125	30
2028	N-Nitrosodiphenylamine	330	µg/kg	21	µg/kg	54	112	30	10	169	30
2024	N-Nitrosodi-n-propylamine	330	µg/kg	23	µg/kg	40	114	30	30	121	30
3597	2,2'-oxybis(1-Chloropropane)	330	µg/kg	26	µg/kg	0	0	0	0	0	0
2118	Pentachlorophenol	330	µg/kg	82	µg/kg	10	110	30	10	182	30
2154	Phenanthrene	330	µg/kg	2	µg/kg	54	110	30	10	200	30
2155	Phenol	330	µg/kg	25	µg/kg	39	110	30	10	144	30
2252	Pyrene	330	µg/kg	1.1	µg/kg	58	113	30	10	200	30
2555	2,4,5-Trichlorophenol	330	µg/kg	25	µg/kg	42	110	30	32	112	30
2559	2,4,6-Trichlorophenol	330	µg/kg	21	µg/kg	37	110	30	22	110	30
1425	2-Fluorobiphenyl					34	110	0	34	110	0
1426	2-Fluorophenol					26	110	0	26	110	0
2512	2,4,6-Tribromophenol					10	118	0	10	118	0
2736	Nitrobenzene-d5					24	112	0	24	112	0
2737	Phenol-d5					28	110	0	28	110	0
2738	Terphenyl-d14					41	119	0	41	119	0

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
2082	Aroclor 1016	33	µg/kg	11	µg/kg	41	130	30	10	200	30
2085	Aroclor 1221	33	µg/kg	13	µg/kg	0	0	0	0	0	0
2088	Aroclor 1232	33	µg/kg	12	µg/kg	0	0	0	0	0	0
2091	Aroclor 1242	33	µg/kg	14	µg/kg	0	0	0	0	0	0
2094	Aroclor 1248	33	µg/kg	15	µg/kg	0	0	0	0	0	0
2097	Aroclor 1254	33	µg/kg	8.8	µg/kg	0	0	0	0	0	0
2100	Aroclor 1260	33	µg/kg	9.8	µg/kg	42	130	30	10	200	30
2732	Decachlorobiphenyl					40	138	0	40	138	0
2739	Tetrachloro-m-xylene					10	127	0	10	127	0
128	Antimony	6	mg/kg	0.33	mg/kg	80	120	20	75	125	20
140	Arsenic	30	mg/kg	0.34	mg/kg	80	120	20	75	125	20
194	Barium	20	mg/kg	0.2	mg/kg	80	120	20	75	125	20
222	Beryllium	0.5	mg/kg	0.029	mg/kg	80	120	20	75	125	20
313	Boron	20	mg/kg	0.65	mg/kg	80	120	20	75	125	20
411	Cadmium	0.5	mg/kg	0.027	mg/kg	80	120	20	75	125	20
413	Calcium	500	mg/kg	8.4	mg/kg	80	120	20	75	125	20
2952	Chromium	1	mg/kg	0.13	mg/kg	80	120	20	75	125	20
637	Cobalt	5	mg/kg	0.34	mg/kg	80	120	20	75	125	20
643	Copper	2.5	mg/kg	0.33	mg/kg	80	120	20	75	125	20
1539	Iron	10	mg/kg	8.7	mg/kg	73	137	20	75	125	20
1605	Lead	10	mg/kg	0.24	mg/kg	80	120	20	75	125	20
1618	Magnesium	500	mg/kg	2.1	mg/kg	80	120	20	75	125	20
1659	Manganese	1.5	mg/kg	0.042	mg/kg	80	120	20	75	125	20
1906	Molybdenum	4	mg/kg	0.3	mg/kg	80	120	20	75	125	20

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1956	Nickel	4	mg/kg	0.28	mg/kg	80	120	20	75	125	20
2214	Potassium	500	mg/kg	3.1	mg/kg	80	120	20	75	125	20
2281	Selenium	25	mg/kg	0.3	mg/kg	80	120	20	75	125	20
2285	Silver	1	mg/kg	0.29	mg/kg	80	120	20	75	125	20
2315	Sodium	500	mg/kg	33	mg/kg	80	120	20	75	125	20
2477	Thallium	200	mg/kg	0.53	mg/kg	80	120	20	75	125	20
2479	Tin	10	mg/kg	0.37	mg/kg	80	120	20	75	125	20
2482	Titanium	5	mg/kg	0.18	mg/kg	80	120	20	75	125	20
2607	Vanadium	5	mg/kg	0.097	mg/kg	80	120	20	75	125	20
2649	Zinc	2	mg/kg	0.56	mg/kg	80	120	20	75	125	20
128	Antimony	0.2	mg/kg	0.013	mg/kg	75	110	20	70	130	20
140	Arsenic	0.5	mg/kg	0.03	mg/kg	74	110	20	70	130	20
194	Barium	0.1	mg/kg	0.052	mg/kg	74	110	20	70	130	20
222	Beryllium	0.1	mg/kg	0.011	mg/kg	80	120	20	70	130	20
411	Cadmium	0.1	mg/kg	0.0071	mg/kg	75	110	20	70	130	20
2952	Chromium	0.2	mg/kg	0.017	mg/kg	75	110	20	70	130	20
637	Cobalt	0.1	mg/kg	0.0024	mg/kg	80	120	20	70	130	20
643	Copper	0.2	mg/kg	0.06	mg/kg	76	110	20	70	130	20
1605	Lead	0.1	mg/kg	0.014	mg/kg	75	110	20	70	130	20
1659	Manganese	0.1	mg/kg	0.025	mg/kg	80	120	20	70	130	20
1906	Molybdenum	0.2	mg/kg	0.036	mg/kg	72	112	20	70	130	20
1956	Nickel	0.1	mg/kg	0.019	mg/kg	77	110	20	70	130	20
2281	Selenium	0.5	mg/kg	0.076	mg/kg	73	110	20	70	130	20
2285	Silver	0.1	mg/kg	0.023	mg/kg	71	110	20	70	130	20
2353	Strontium	1	mg/kg	0.081	mg/kg	74	110	20	70	130	20
2477	Thallium	0.1	mg/kg	0.0022	mg/kg	75	110	20	70	130	20
2479	Tin	1	mg/kg	0.026	mg/kg	60	125	20	70	130	20
2602	Tungsten	1	mg/kg	0.032	mg/kg	75	125	20	80	120	20
2607	Vanadium	0.5	mg/kg	0.034	mg/kg	76	110	20	70	130	20
2649	Zinc	1	mg/kg	0.39	mg/kg	73	114	20	70	130	20
2651	Zirconium	2	mg/kg	0.2	mg/kg	80	120	20	80	120	20

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
88	Aluminum	50	µg/L	2.6	µg/L	70	118	20	70	130	20
128	Antimony	2	µg/L	0.065	µg/L	62	110	20	70	130	20
140	Arsenic	5	µg/L	0.47	µg/L	82	120	20	70	130	20
194	Barium	1	µg/L	0.13	µg/L	70	110	20	70	130	20
222	Beryllium	1	µg/L	0.11	µg/L	86	113	20	70	130	20
411	Cadmium	1	µg/L	0.086	µg/L	82	116	20	70	130	20
2952	Chromium	2	µg/L	0.92	µg/L	69	114	20	70	130	20
637	Cobalt	1	µg/L	0.034	µg/L	70	110	20	70	130	20
643	Copper	2	µg/L	0.51	µg/L	73	111	20	70	130	20
1539	Iron	20	µg/L	12	µg/L	72	115	20	70	130	20
1605	Lead	1	µg/L	0.11	µg/L	69	110	20	70	130	20
1659	Manganese	1	µg/L	0.3	µg/L	75	110	20	70	130	20
1906	Molybdenum	2	µg/L	0.27	µg/L	75	125	20	70	130	20
1956	Nickel	2	µg/L	0.23	µg/L	70	112	20	70	130	20
2281	Selenium	5	µg/L	0.59	µg/L	90	131	20	70	130	20
2285	Silver	1	µg/L	0.047	µg/L	70	115	20	70	130	20
2353	Strontium	10	µg/L	0.35	µg/L	80	120	20	70	130	20
2477	Thallium	1	µg/L	0.022	µg/L	69	114	20	70	130	20
2479	Tin	10	µg/L	0.3	µg/L	80	120	20	70	130	20
2602	Tungsten	10	µg/L	0.16	µg/L	80	120	20	80	120	20
2607	Vanadium	20	µg/L	0.42	µg/L	77	110	20	70	130	20
2649	Zinc	20	µg/L	2.3	µg/L	90	127	20	70	130	20
2651	Zirconium	20	µg/L	1.3	µg/L	80	120	20	80	120	20
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STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
88	Aluminum	200	µg/L	47	µg/L	80	120	20	75	125	20
128	Antimony	60	µg/L	4.1	µg/L	80	120	20	75	125	20
140	Arsenic	300	µg/L	4.3	µg/L	80	120	20	75	125	20
194	Barium	200	µg/L	3.2	µg/L	80	120	20	75	125	20
222	Beryllium	5	µg/L	0.3	µg/L	80	120	20	75	125	20
313	Boron	200	µg/L	16	µg/L	80	120	20	75	125	20
411	Cadmium	5	µg/L	0.42	µg/L	80	120	20	75	125	20
413	Calcium	5000	µg/L	80	µg/L	80	120	20	75	125	20
2952	Chromium	10	µg/L	1.6	µg/L	80	120	20	75	125	20
637	Cobalt	50	µg/L	1.2	µg/L	80	120	20	75	125	20
643	Copper	25	µg/L	1.8	µg/L	80	120	20	75	125	20
1539	Iron	100	µg/L	32	µg/L	77	127	20	75	125	20
1605	Lead	100	µg/L	1.7	µg/L	80	120	20	75	125	20
1618	Magnesium	5000	µg/L	86	µg/L	80	120	20	75	125	20
1659	Manganese	15	µg/L	0.23	µg/L	80	120	20	75	125	20
1906	Molybdenum	40	µg/L	2.3	µg/L	80	120	20	75	125	20
1956	Nickel	40	µg/L	1.4	µg/L	80	120	20	75	125	20
2214	Potassium	5000	µg/L	54	µg/L	80	120	20	75	125	20
2281	Selenium	250	µg/L	2.4	µg/L	80	120	20	75	125	20
2285	Silver	10	µg/L	2.1	µg/L	80	120	20	75	125	20
2315	Sodium	5000	µg/L	410	µg/L	80	120	20	75	125	20
2477	Thallium	200	µg/L	4.7	µg/L	80	120	20	75	125	20
2479	Tin	100	µg/L	5.2	µg/L	80	120	20	75	125	20
2482	Titanium	50	µg/L	1.9	µg/L	80	120	20	75	125	20
2607	Vanadium	50	µg/L	1.9	µg/L	80	120	20	75	125	20
2649	Zinc	20	µg/L	6.6	µg/L	80	120	20	75	125	20

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
2082	Aroclor 1016	1	µg/L	0.25	µg/L	50	115	30	10	200	30
2085	Aroclor 1221	1	µg/L	0.49	µg/L	0	0	0	0	0	0
2088	Aroclor 1232	1	µg/L	0.41	µg/L	0	0	0	0	0	0
2091	Aroclor 1242	1	µg/L	0.11	µg/L	0	0	0	0	0	0
2094	Aroclor 1248	1	µg/L	0.049	µg/L	0	0	0	0	0	0
2097	Aroclor 1254	1	µg/L	0.087	µg/L	0	0	0	0	0	0
2100	Aroclor 1260	1	µg/L	0.071	µg/L	45	112	30	10	150	30
2732	Decachlorobiphenyl					10	110	0	10	110	0
2739	Tetrachloro-m-xylene					35	130	0	35	130	0
1	Acenaphthene	10	µg/L	0.054	µg/L	40	110	30	36	110	30
5	Acenaphthylene	10	µg/L	0.054	µg/L	43	110	30	39	110	30
24	Acetophenone	10	µg/L	0.55	µg/L	0	0	0	0	0	0
122	Anthracene	10	µg/L	0.054	µg/L	54	114	30	46	110	30
158	Atrazine	10	µg/L	0.65	µg/L	0	0	0	0	0	0
3398	Benzaldehyde	10	µg/L	0.75	µg/L	0	0	0	0	0	0
202	Benzo(a)anthracene	10	µg/L	0.052	µg/L	55	115	30	52	110	30
205	Benzo(b)fluoranthene	10	µg/L	0.049	µg/L	43	122	30	33	114	30
208	Benzo(k)fluoranthene	10	µg/L	0.049	µg/L	43	124	30	32	121	30
210	Benzo(ghi)perylene	10	µg/L	0.053	µg/L	45	120	30	34	116	30
211	Benzo(a)pyrene	10	µg/L	0.048	µg/L	43	116	30	33	110	30
3474	1,1'-Biphenyl	10	µg/L	0.55	µg/L	0	0	0	0	0	0
289	bis(2-Chloroethoxy)methane	10	µg/L	0.49	µg/L	39	110	30	35	110	30
293	bis(2-Chloroethyl) ether	10	µg/L	0.088	µg/L	34	113	30	27	110	30
302	bis(2-Ethylhexyl) phthalate	10	µg/L	0.88	µg/L	36	163	30	40	140	30
348	4-Bromophenyl phenyl ether	10	µg/L	0.52	µg/L	51	114	30	42	113	30
403	Butyl benzyl phthalate	10	µg/L	0.51	µg/L	53	126	30	51	121	30
5101	Caprolactam	10	µg/L	0.61	µg/L	0	0	0	0	0	0
2751	Carbazole	10	µg/L	0.54	µg/L	53	120	30	49	114	30
518	4-Chloroaniline	10	µg/L	0.56	µg/L	10	110	30	10	110	30
578	4-Chloro-3-methylphenol	10	µg/L	0.41	µg/L	39	110	30	33	110	30
589	2-Chloronaphthalene	10	µg/L	0.62	µg/L	39	110	30	34	110	30

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
600	2-Chlorophenol	10	µg/L	1.1	µg/L	27	110	30	26	110	30
602	4-Chlorophenyl phenyl ether	10	µg/L	0.55	µg/L	50	115	30	43	113	30
633	Chrysene	10	µg/L	0.048	µg/L	55	115	30	52	111	30
860	Dibenz(a,h)anthracene	10	µg/L	0.039	µg/L	46	122	30	35	118	30
863	Dibenzofuran	10	µg/L	0.54	µg/L	46	111	30	41	110	30
891	Di-n-butyl phthalate	10	µg/L	0.61	µg/L	55	122	30	50	117	30
918	3,3'-Dichlorobenzidine	50	µg/L	0.48	µg/L	19	110	30	10	110	30
971	2,4-Dichlorophenol	10	µg/L	1.1	µg/L	33	110	30	30	110	30
1082	Diethyl phthalate	10	µg/L	0.63	µg/L	33	134	30	33	130	30
1145	2,4-Dimethylphenol	10	µg/L	0.56	µg/L	12	110	30	11	110	30
1149	Dimethyl phthalate	10	µg/L	0.44	µg/L	15	143	30	36	124	30
1167	4,6-Dinitro-2-methylphenol	50	µg/L	0.27	µg/L	28	112	30	25	110	30
1187	2,4-Dinitrophenol	50	µg/L	3.5	µg/L	17	112	30	11	119	30
1191	2,4-Dinitrotoluene	10	µg/L	0.4	µg/L	52	123	30	46	119	30
1193	2,6-Dinitrotoluene	10	µg/L	0.47	µg/L	52	119	30	48	115	30
1162	Di-n-octyl phthalate	10	µg/L	0.39	µg/L	44	128	30	36	124	30
1414	Fluoranthene	10	µg/L	0.036	µg/L	54	122	30	53	111	30
1417	Fluorene	10	µg/L	0.043	µg/L	47	112	30	43	110	30
1482	Hexachlorobenzene	10	µg/L	0.065	µg/L	51	112	30	40	113	30
1489	Hexachlorobutadiene	10	µg/L	0.51	µg/L	13	110	30	14	110	30
1492	Hexachlorocyclopentadiene	50	µg/L	0.74	µg/L	10	110	30	10	110	30
1497	Hexachloroethane	10	µg/L	0.58	µg/L	12	110	30	10	110	30
1535	Indeno(1,2,3-cd)pyrene	10	µg/L	0.065	µg/L	46	121	30	36	116	30
1566	Isophorone	10	µg/L	0.5	µg/L	44	128	30	34	125	30
1829	2-Methylnaphthalene	10	µg/L	0.061	µg/L	35	110	30	35	110	30
1851	2-Methylphenol	10	µg/L	0.56	µg/L	30	110	30	26	110	30
1857	4-Methylphenol	10	µg/L	0.64	µg/L	32	110	30	25	110	30
1932	Naphthalene	10	µg/L	0.069	µg/L	31	110	30	32	110	30
1960	2-Nitroaniline	50	µg/L	0.43	µg/L	43	130	30	31	129	30
1964	3-Nitroaniline	50	µg/L	0.67	µg/L	45	116	30	23	112	30
1968	4-Nitroaniline	50	µg/L	0.47	µg/L	45	120	30	26	115	30

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
1972	Nitrobenzene	10	µg/L	0.053	µg/L	37	115	30	26	118	30
1998	2-Nitrophenol	10	µg/L	1.3	µg/L	29	110	30	30	110	30
2001	4-Nitrophenol	50	µg/L	0.63	µg/L	12	130	30	13	127	30
2028	N-Nitrosodiphenylamine	10	µg/L	0.46	µg/L	53	113	30	28	118	30
2024	N-Nitrosodi-n-propylamine	10	µg/L	0.53	µg/L	37	121	30	25	119	30
3597	2,2'-oxybis(1-Chloropropane)	10	µg/L	0.52	µg/L	0	0	0	0	0	0
2118	Pentachlorophenol	10	µg/L	0.48	µg/L	26	110	30	23	110	30
2154	Phenanthrene	10	µg/L	0.087	µg/L	52	114	30	47	110	30
2155	Phenol	10	µg/L	0.96	µg/L	14	112	30	16	110	30
2252	Pyrene	10	µg/L	0.048	µg/L	55	120	30	54	115	30
2555	2,4,5-Trichlorophenol	10	µg/L	0.96	µg/L	39	110	30	36	110	30
2559	2,4,6-Trichlorophenol	10	µg/L	1.4	µg/L	35	110	30	34	110	30
1425	2-Fluorobiphenyl					28	110	0	28	110	0
1426	2-Fluorophenol					10	110	0	10	110	0
2512	2,4,6-Tribromophenol					22	120	0	22	120	0
2736	Nitrobenzene-d5					27	111	0	27	111	0
2737	Phenol-d5					10	110	0	10	110	0
2738	Terphenyl-d14					37	119	0	37	119	0
11	Acetone	10	µg/L	0.74	µg/L	22	200	95	45	128	30
196	Benzene	1	µg/L	0.22	µg/L	80	116	20	78	118	20
323	Bromodichloromethane	1	µg/L	0.14	µg/L	87	130	30	80	146	30
340	Bromoform	1	µg/L	0.17	µg/L	76	150	30	58	176	30
343	Bromomethane	1	µg/L	0.36	µg/L	64	129	30	55	145	30
372	2-Butanone	10	µg/L	0.39	µg/L	28	237	65	71	123	30
459	Carbon disulfide	1	µg/L	0.28	µg/L	73	139	30	69	138	41
463	Carbon tetrachloride	1	µg/L	0.19	µg/L	75	149	30	63	176	30
521	Chlorobenzene	1	µg/L	0.2	µg/L	76	117	20	76	117	20
535	Dibromochloromethane	1	µg/L	0.19	µg/L	81	138	30	71	158	30
550	Chloroethane	1	µg/L	0.24	µg/L	66	126	30	59	142	30
569	Chloroform	1	µg/L	0.16	µg/L	84	128	30	83	141	30
574	Chloromethane	1	µg/L	0.14	µg/L	48	123	30	40	137	39

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
669	Cyclohexane	1	µg/L	0.12	µg/L	70	130	30	70	130	30
539	1,2-Dibromo-3-chloropropane	2	µg/L	0.28	µg/L	70	130	30	70	130	30
870	1,2-Dibromoethane	1	µg/L	0.24	µg/L	70	130	30	70	130	30
904	1,2-Dichlorobenzene	1	µg/L	0.2	µg/L	70	130	30	70	130	30
907	1,3-Dichlorobenzene	1	µg/L	0.18	µg/L	70	130	30	70	130	30
910	1,4-Dichlorobenzene	1	µg/L	0.22	µg/L	70	130	30	70	130	30
924	Dichlorodifluoromethane	1	µg/L	0.25	µg/L	70	130	30	70	130	30
933	1,1-Dichloroethane	1	µg/L	0.21	µg/L	86	123	30	88	127	30
936	1,2-Dichloroethane	1	µg/L	0.16	µg/L	79	136	30	71	160	30
948	cis-1,2-Dichloroethene	1	µg/L	0.21	µg/L	85	113	30	87	114	30
950	trans-1,2-Dichloroethene	1	µg/L	0.16	µg/L	80	120	30	85	116	30
943	1,1-Dichloroethene	1	µg/L	0.18	µg/L	63	130	20	62	130	20
986	1,2-Dichloropropane	1	µg/L	0.15	µg/L	82	115	30	87	114	30
998	cis-1,3-Dichloropropene	1	µg/L	0.12	µg/L	84	130	30	82	130	30
1000	trans-1,3-Dichloropropene	1	µg/L	0.17	µg/L	84	130	30	73	147	30
1332	Ethylbenzene	1	µg/L	0.19	µg/L	86	116	30	86	132	30
1515	2-Hexanone	10	µg/L	0.35	µg/L	35	200	52	81	128	30
1578	Isopropylbenzene	1	µg/L	0.15	µg/L	70	130	30	70	130	30
1774	Methyl acetate	10	µg/L	0.52	µg/L	70	130	30	70	130	30
1799	Methylcyclohexane	1	µg/L	0.5	µg/L	70	130	30	70	130	30
1811	Methylene chloride	1	µg/L	0.19	µg/L	78	118	30	82	115	30
1845	4-Methyl-2-pentanone	10	µg/L	0.32	µg/L	78	141	32	82	135	30
2772	Methyl tert-butyl ether	5	µg/L	0.18	µg/L	70	130	30	70	130	30
2355	Styrene	1	µg/L	0.13	µg/L	85	117	30	83	120	30
2439	1,1,2,2-Tetrachloroethane	1	µg/L	0.22	µg/L	85	118	30	88	116	30
2445	Tetrachloroethene	1	µg/L	0.19	µg/L	88	113	30	85	121	30
2489	Toluene	1	µg/L	0.17	µg/L	74	119	20	70	119	20
2515	1,2,4-Trichlorobenzene	1	µg/L	0.19	µg/L	70	130	30	70	130	30
2518	1,1,1-Trichloroethane	1	µg/L	0.21	µg/L	78	140	30	71	162	30
2522	1,1,2-Trichloroethane	1	µg/L	0.22	µg/L	83	122	30	86	129	30
2525	Trichloroethene	1	µg/L	0.28	µg/L	75	122	20	62	130	20

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
1428	Trichlorofluoromethane	1	µg/L	0.16	µg/L	70	130	30	70	130	30
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	1	µg/L	0.26	µg/L	70	130	30	70	130	30
2613	Vinyl chloride	1	µg/L	0.21	µg/L	61	120	30	88	126	30
2627	Xylenes (total)	2	µg/L	0.44	µg/L	87	116	30	89	121	30
337	4-Bromofluorobenzene					74	116	0	74	116	0
2735	1,2-Dichloroethane-d4					61	128	0	61	128	0
2740	Toluene-d8					76	110	0	76	110	0
2863	Dibromofluoromethane					73	122	0	73	122	0
1701	Mercury	0.2	µg/L	0.09	µg/L	82	131	20	68	149	20
1701	Mercury	0.1	mg/kg	0.013	mg/kg	73	123	20	10	199	50
667	Total Cyanide	0.5	mg/kg	0.11	mg/kg	68	123	20	50	134	20
667	Total Cyanide	0.01	mg/L	0.0034	mg/L	69	118	20	42	140	20
3041	Total Alkalinity	5	mg/L	0.71	mg/L	90	127	20	10	160	24
512	Chloride	1	mg/L	0.1	mg/L	90	110	20	80	120	20
3744	Nitrate as N	0.1	mg/L	0.031	mg/L	90	110	20	80	120	20
3746	Nitrite as N	0.1	mg/L	0.022	mg/L	90	110	20	80	120	20
2363	Sulfate	1	mg/L	0.12	mg/L	90	110	20	80	120	20
3655	Dissolved Organic Carbon	1	mg/L	0.29	mg/L	88	115	20	72	136	20
1469	Hardness, as CaCO3	33.075	mg/L	33.075	mg/L	0	0	0	0	0	0
3693	Acid-soluble sulfide	1	mg/L	0.86	mg/L	75	125	20	75	125	20

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
3443	Acetylene	1	µg/L	0.19	µg/L	70	130	30	70	130	30
3377	Ethane	0.5	µg/L	0.16	µg/L	74	138	30	74	138	30
3386	Ethene	0.5	µg/L	0.16	µg/L	73	140	30	73	140	30
2841	Methane	0.5	µg/L	0.11	µg/L	75	127	30	75	127	30
196	Benzene	0.025	mg/L	0.23	mg/L	76	118	30	76	117	30
3271	2-Butanone (MEK)	0.05	mg/L	1	mg/L	0	0	0	0	0	0
463	Carbon tetrachloride	0.025	mg/L	0.45	mg/L	71	124	30	72	124	30
521	Chlorobenzene	0.025	mg/L	0.28	mg/L	76	113	30	72	114	30
569	Chloroform	0.025	mg/L	0.4	mg/L	82	117	30	82	117	30
936	1,2-Dichloroethane	0.025	mg/L	0.48	mg/L	78	122	30	80	120	30
946	1,1-Dichloroethylene	0.07	mg/L	0.6	mg/L	0	0	0	0	0	0
2446	Tetrachloroethylene	0.07	mg/L	0.83	mg/L	0	0	0	0	0	0
2526	Trichloroethylene	0.05	mg/L	0.41	mg/L	0	0	0	0	0	0
2613	Vinyl chloride	0.025	mg/L	0.44	mg/L	47	123	30	54	118	30
337	4-Bromofluorobenzene					84	125	0	84	125	0
2735	1,2-Dichloroethane-d4					80	122	0	80	122	0
2740	Toluene-d8					90	122	0	90	122	0
2863	Dibromofluoromethane					86	124	0	86	125	0

STL NORTH CANTON - SUMMARY REFERENCE DATA
REMEDIAL INVESTIGATION AND FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL SITE
MORAINE, OHIO

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
2778	m-Cresol & p-Cresol	0.04	mg/L	0.75	mg/L	27	110	30	46	109	32
910	1,4-Dichlorobenzene	0.004	mg/L	0.52	mg/L	16	110	30	18	110	36
1191	2,4-Dinitrotoluene	0.02	mg/L	0.4	mg/L	45	126	30	31	131	32
1482	Hexachlorobenzene	0.02	mg/L	0.065	mg/L	47	116	30	36	132	22
1489	Hexachlorobutadiene	0.02	mg/L	0.51	mg/L	10	110	30	18	116	32
1497	Hexachloroethane	0.02	mg/L	0.58	mg/L	10	110	30	18	110	33
1853	o-Cresol	0.004	mg/L	0.56	mg/L	24	110	30	33	115	31
1972	Nitrobenzene	0.004	mg/L	0.053	mg/L	35	117	30	19	211	59
2118	Pentachlorophenol	0.04	mg/L	0.48	mg/L	12	110	30	10	140	56
2256	Pyridine	0.02	mg/L	0.78	mg/L	10	110	30	10	148	65
2555	2,4,5-Trichlorophenol	0.02	mg/L	0.96	mg/L	35	111	30	24	143	22
2559	2,4,6-Trichlorophenol	0.02	mg/L	1.4	mg/L	32	110	30	36	135	27
1425	2-Fluorobiphenyl					22	110	0	22	110	0
1426	2-Fluorophenol					10	110	0	10	110	0
2512	2,4,6-Tribromophenol					17	117	0	17	117	0
2736	Nitrobenzene-d5					29	111	0	29	111	0
2737	Phenol-d5					10	110	0	10	110	0
2738	Terphenyl-d14					40	119	0	40	119	0

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#	*Compound*	*RL*	*Units*	*MDL*	*Units*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
140	Arsenic	0.5	mg/L	0.0043	mg/L	50	150	20	50	150	20
194	Barium	10	mg/L	0.0032	mg/L	50	150	20	50	150	20
411	Cadmium	0.1	mg/L	0.00042	mg/L	50	150	20	50	150	20
2952	Chromium	0.5	mg/L	0.0016	mg/L	50	150	20	50	150	20
1605	Lead	0.5	mg/L	0.0017	mg/L	50	150	20	50	150	20
2281	Selenium	0.25	mg/L	0.0024	mg/L	50	150	20	50	150	20
2285	Silver	0.5	mg/L	0.0021	mg/L	50	150	20	50	150	20
1701	Mercury	0.002	mg/L	0.00009	mg/L	50	150	20	50	150	20
233	Lindane	0.0005	mg/L	0.0062	mg/L	56	110	88	50	150	50
476	Chlordane (technical)	0.005	mg/L	0.075	mg/L	0	0	0	0	0	0
1270	Endrin	0.0005	mg/L	0.0074	mg/L	50	110	93	50	150	50
1470	Heptachlor	0.0005	mg/L	0.0062	mg/L	57	110	88	50	150	50
1479	Heptachlor epoxide	0.0005	mg/L	0.0065	mg/L	56	110	86	50	150	50
1741	Methoxychlor	0.001	mg/L	0.01	mg/L	41	126	94	50	150	50
2499	Toxaphene	0.02	mg/L	0.33	mg/L	0	0	0	0	0	0
2732	Decachlorobiphenyl					31	115	0	31	115	0
2739	Tetrachloro-m-xylene					47	110	0	47	110	0
690	2,4-D	0.5	mg/L	1.5	mg/L	35	136	50	54	114	67
2291	2,4,5-TP (Silvex)	0.1	mg/L	0.16	mg/L	0	0	0	0	0	0
2924	2,4-Dichlorophenylacetic acid					37	116	0	37	116	0
2673	Flashpoint		deg F		deg F	75	125	20	75	125	20
645	Corrosivity	-	No Units		No Units	97	103	20	97	103	20
666	Reactive Cyanide	200	mg/kg	71	mg/kg	10	200	100	10	200	100
2365	Reactive Sulfide	500	mg/kg	61	mg/kg	10	200	100	10	200	100

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#	*Compound*	*RL*	*Units*	*MDL*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
3443	Acetylene	1	µg/L	0.19	70	130	30	70	130	30
3377	Ethane	0.5	µg/L	0.16	74	138	30	74	138	30
3386	Ethene	0.5	µg/L	0.16	73	140	30	73	140	30
2841	Methane	0.5	µg/L	0.11	75	127	30	75	127	30
196	Benzene	0.025	mg/L	0.23	76	118	30	76	117	30
3271	2-Butanone (MEK)	0.05	mg/L	1	0	0	0	0	0	0
463	Carbon tetrachloride	0.025	mg/L	0.45	71	124	30	72	124	30
521	Chlorobenzene	0.025	mg/L	0.28	76	113	30	72	114	30
569	Chloroform	0.025	mg/L	0.4	82	117	30	82	117	30
936	1,2-Dichloroethane	0.025	mg/L	0.48	78	122	30	80	120	30
946	1,1-Dichloroethylene	0.07	mg/L	0.6	0	0	0	0	0	0
2446	Tetrachloroethylene	0.07	mg/L	0.83	0	0	0	0	0	0
2526	Trichloroethylene	0.05	mg/L	0.41	0	0	0	0	0	0
2613	Vinyl chloride	0.025	mg/L	0.44	47	123	30	54	118	30
337	4-Bromofluorobenzene				84	125	0	84	125	0
2735	1,2-Dichloroethane-d4				80	122	0	80	122	0
2740	Toluene-d8				90	122	0	90	122	0
2863	Dibromofluoromethane				86	124	0	86	125	0
2778	m-Cresol & p-Cresol	0.04	mg/L	0.75	27	110	30	46	109	32
910	1,4-Dichlorobenzene	0.004	mg/L	0.52	16	110	30	18	110	36
1191	2,4-Dinitrotoluene	0.02	mg/L	0.4	45	126	30	31	131	32
1482	Hexachlorobenzene	0.02	mg/L	0.065	47	116	30	36	132	22
1489	Hexachlorobutadiene	0.02	mg/L	0.51	10	110	30	18	116	32
1497	Hexachloroethane	0.02	mg/L	0.58	10	110	30	18	110	33
1853	o-Cresol	0.004	mg/L	0.56	24	110	30	33	115	31
1972	Nitrobenzene	0.004	mg/L	0.053	35	117	30	19	211	59
2118	Pentachlorophenol	0.04	mg/L	0.48	12	110	30	10	140	56
2256	Pyridine	0.02	mg/L	0.78	10	110	30	10	148	65
2555	2,4,5-Trichlorophenol	0.02	mg/L	0.96	35	111	30	24	143	22

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#	*Compound*	*RL*	*Units*	*MDL*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
2559	2,4,6-Trichlorophenol	0.02	mg/L	1.4	32	110	30	36	135	27
1425	2-Fluorobiphenyl				22	110	0	22	110	0
1426	2-Fluorophenol				10	110	0	10	110	0
2512	2,4,6-Tribromophenol				17	117	0	17	117	0
2736	Nitrobenzene-d5				29	111	0	29	111	0
2737	Phenol-d5				10	110	0	10	110	0
2738	Terphenyl-d14				40	119	0	40	119	0
140	Arsenic	0.5	mg/L	0.0043	50	150	20	50	150	20
194	Barium	10	mg/L	0.0032	50	150	20	50	150	20
411	Cadmium	0.1	mg/L	0.00042	50	150	20	50	150	20
2952	Chromium	0.5	mg/L	0.0016	50	150	20	50	150	20
1605	Lead	0.5	mg/L	0.0017	50	150	20	50	150	20
2281	Selenium	0.25	mg/L	0.0024	50	150	20	50	150	20
2285	Silver	0.5	mg/L	0.0021	50	150	20	50	150	20
1701	Mercury	0.002	mg/L	0.00009	50	150	20	50	150	20
233	Lindane	0.0005	mg/L	0.0062	56	110	88	50	150	50
476	Chlordane (technical)	0.005	mg/L	0.075	0	0	0	0	0	0
1270	Endrin	0.0005	mg/L	0.0074	50	110	93	50	150	50
1470	Heptachlor	0.0005	mg/L	0.0062	57	110	88	50	150	50
1479	Heptachlor epoxide	0.0005	mg/L	0.0065	56	110	86	50	150	50
1741	Methoxychlor	0.001	mg/L	0.01	41	126	94	50	150	50
2499	Toxaphene	0.02	mg/L	0.33	0	0	0	0	0	0
2732	Decachlorobiphenyl				31	115	0	31	115	0
2739	Tetrachloro-m-xylene				47	110	0	47	110	0
690	2,4-D	0.5	mg/L	1.5	35	136	50	54	114	67
2291	2,4,5-TP (Silvex)	0.1	mg/L	0.16	0	0	0	0	0	0
2924	2,4-Dichlorophenylacetic acid				37	116	0	37	116	0

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#	*Compound*	*RL*	*Units*	*MDL*	*LCL*	*UCL*	*RPD*	*LCL*	*UCL*	*RPD*
2673	Flashpoint		deg F		75	125	20	75	125	20
645	Corrosivity	-	No Units		97	103	20	97	103	20
666	Reactive Cyanide	200	mg/kg	71	10	200	100	10	200	100
2365	Reactive Sulfide	500	mg/kg	61	10	200	100	10	200	100

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
Volatile Organics - Water Samples												
11	Acetone	10	ug/L	1.1	20	22	200	95	10	45	128	30
196	Benzene	1	ug/L	0.13	20	80	116	20	10	78	118	20
323	Bromodichloromethane	1	ug/L	0.15	20	87	130	30	10	80	146	30
340	Bromoform	1	ug/L	0.64	20	76	150	30	10	58	176	30
343	Bromomethane	1	ug/L	0.41	20	64	129	30	10	55	145	30
372	2-Butanone	10	ug/L	0.57	20	28	237	65	10	71	123	30
459	Carbon disulfide	1	ug/L	0.13	20	73	139	30	10	69	138	41
463	Carbon tetrachloride	1	ug/L	0.13	20	75	149	30	10	63	176	30
521	Chlorobenzene	1	ug/L	0.15	20	76	117	20	10	76	117	20
535	Dibromochloromethane	1	ug/L	0.18	20	81	138	30	10	71	158	30
550	Chloroethane	1	ug/L	0.29	20	66	126	30	10	59	142	30
569	Chloroform	1	ug/L	0.16	20	84	128	30	10	83	141	30
574	Chloromethane	1	ug/L	0.3	20	48	123	30	10	40	137	39
669	Cyclohexane	1	ug/L	0.12	20	70	130	30	10	70	130	30
539	1,2-Dibromo-3-chloropropane	2	ug/L	0.67	20	70	130	30	10	70	130	30
870	1,2-Dibromoethane	1	ug/L	0.24	20	70	130	30	10	70	130	30
904	1,2-Dichlorobenzene	1	ug/L	0.13	20	70	130	30	10	70	130	30
907	1,3-Dichlorobenzene	1	ug/L	0.14	20	70	130	30	10	70	130	30
910	1,4-Dichlorobenzene	1	ug/L	0.13	20	70	130	30	10	70	130	30
924	Dichlorodifluoromethane	1	ug/L	0.31	20	70	130	30	10	70	130	30
933	1,1-Dichloroethane	1	ug/L	0.15	20	86	123	30	10	88	127	30
936	1,2-Dichloroethane	1	ug/L	0.22	20	79	136	30	10	71	160	30
948	cis-1,2-Dichloroethene	1	ug/L	0.17	20	85	113	30	10	87	114	30
950	trans-1,2-Dichloroethene	1	ug/L	0.19	20	80	120	30	10	85	116	30
943	1,1-Dichloroethene	1	ug/L	0.19	20	63	130	20	10	62	130	20
986	1,2-Dichloropropane	1	ug/L	0.18	20	82	115	30	10	87	114	30
998	cis-1,3-Dichloropropene	1	ug/L	0.14	20	84	130	30	10	82	130	30
1000	trans-1,3-Dichloropropene	1	ug/L	0.19	20	84	130	30	10	73	147	30
1332	Ethylbenzene	1	ug/L	0.17	20	86	116	30	10	86	132	30
1515	2-Hexanone	10	ug/L	0.41	20	35	200	52	10	81	128	30
1578	Isopropylbenzene	1	ug/L	0.13	20	70	130	30	10	70	130	30
1774	Methyl acetate	10	ug/L	0.38	20	70	130	30	10	70	130	30
1799	Methylcyclohexane	1	ug/L	0.13	20	70	130	30	10	70	130	30
1811	Methylene chloride	1	ug/L	0.33	20	78	118	30	10	82	115	30
1845	4-Methyl-2-pentanone	10	ug/L	0.32	20	78	141	32	10	82	135	30
2772	Methyl tert-butyl ether	5	ug/L	0.17	20	70	130	30	10	70	130	30
2355	Styrene	1	ug/L	0.11	20	85	117	30	10	83	120	30
2439	1,1,2,2-Tetrachloroethane	1	ug/L	0.18	20	85	118	30	10	88	116	30
2445	Tetrachloroethene	1	ug/L	0.29	20	88	113	30	10	85	121	30
2489	Toluene	1	ug/L	0.13	20	74	119	20	10	70	119	20
2515	1,2,4-Trichlorobenzene	1	ug/L	0.15	20	70	130	30	10	70	130	30
2518	1,1,1-Trichloroethane	1	ug/L	0.22	20	78	140	30	10	71	162	30
2522	1,1,2-Trichloroethane	1	ug/L	0.27	20	83	122	30	10	86	129	30
2525	Trichloroethene	1	ug/L	0.17	20	75	122	20	10	62	130	20
1428	Trichlorofluoromethane	1	ug/L	0.21	20	70	130	30	10	70	130	30
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	1	ug/L	0.28	20	70	130	30	10	70	130	30

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
2613	Vinyl chloride	1	ug/L	0.22	20	61	120	30	10	88	126	30
2627	Xylenes (total)	2	ug/L	0.28	60	87	116	30	30	89	121	30
337	4-Bromofluorobenzene				10	74	116	0	10	74	116	0
2735	1,2-Dichloroethane-d4				10	61	128	0	10	61	128	0
2740	Toluene-d8				10	76	110	0	10	76	110	0
2863	Dibromofluoromethane				10	73	122	0	10	73	122	0
Volatile Organics - Soil/Solid Samples												
11	Acetone	20	ug/kg	6.3	20	58	130	30	50	10	200	66
196	Benzene	5	ug/kg	0.23	20	75	129	20	50	55	138	20
323	Bromodichloromethane	5	ug/kg	0.28	20	72	125	30	50	47	131	51
340	Bromoform	5	ug/kg	0.33	20	43	149	30	50	26	141	64
343	Bromomethane	5	ug/kg	0.54	20	24	152	30	50	15	152	72
372	2-Butanone	20	ug/kg	1.4	20	27	200	46	50	21	195	60
459	Carbon disulfide	5	ug/kg	0.44	20	50	137	30	50	27	149	73
463	Carbon tetrachloride	5	ug/kg	0.37	20	57	137	30	50	32	143	68
521	Chlorobenzene	5	ug/kg	0.33	20	75	127	22	50	49	139	22
535	Dibromochloromethane	5	ug/kg	0.55	20	49	135	30	50	44	135	61
550	Chloroethane	5	ug/kg	0.86	20	31	144	30	50	32	140	66
569	Chloroform	5	ug/kg	0.29	40	73	115	30	50	59	128	46
574	Chloromethane	5	ug/kg	0.41	20	15	136	30	50	28	130	81
669	Cyclohexane	10	ug/kg	0.33	20	50	150	20	50	50	150	20
539	1,2-Dibromo-3-chloropropane	10	ug/kg	1.3	20	50	150	30	50	50	150	20
870	1,2-Dibromoethane	5	ug/kg	0.5	20	50	150	30	50	50	150	20
904	1,2-Dichlorobenzene	5	ug/kg	0.36	20	50	150	30	50	50	150	20
907	1,3-Dichlorobenzene	5	ug/kg	0.35	20	50	150	20	50	50	150	20
910	1,4-Dichlorobenzene	5	ug/kg	0.66	20	50	150	20	50	50	150	20
924	Dichlorodifluoromethane	5	ug/kg	0.5	20	50	150	20	50	50	150	20
933	1,1-Dichloroethane	5	ug/kg	0.36	20	77	119	30	50	56	130	54
936	1,2-Dichloroethane	5	ug/kg	0.34	20	78	121	30	50	56	126	38
948	cis-1,2-Dichloroethene	5	ug/kg	0.36	20	77	114	30	50	48	127	52
950	trans-1,2-Dichloroethene	5	ug/kg	0.41	20	68	117	30	50	47	127	58
943	1,1-Dichloroethene	5	ug/kg	0.52	20	55	142	27	50	43	147	27
986	1,2-Dichloropropane	5	ug/kg	0.69	20	78	116	30	50	54	125	43
998	cis-1,3-Dichloropropene	5	ug/kg	0.34	20	71	125	30	50	30	138	49
1000	trans-1,3-Dichloropropene	5	ug/kg	0.54	20	67	125	30	50	34	134	57
1332	Ethylbenzene	5	ug/kg	0.26	20	79	114	30	50	36	133	72
1515	2-Hexanone	20	ug/kg	0.63	20	29	200	41	50	20	190	70
1578	Isopropylbenzene	5	ug/kg	0.16	20	50	150	20	50	50	150	20
1774	Methyl acetate	10	ug/kg	1.4	20	50	150	20	50	50	150	20
1799	Methylcyclohexane	10	ug/kg	0.31	20	50	150	20	50	50	150	20
1811	Methylene chloride	5	ug/kg	0.67	20	58	130	30	50	45	129	49
1845	4-Methyl-2-pentanone	20	ug/kg	0.54	20	68	142	60	50	42	143	60
2772	Methyl tert-butyl ether	20	ug/kg	0.43	20	70	130	30	50	70	130	30
2355	Styrene	5	ug/kg	0.15	20	80	114	30	50	23	136	65
2439	1,1,2,2-Tetrachloroethane	5	ug/kg	0.34	20	70	133	30	50	33	162	90
2445	Tetrachloroethene	5	ug/kg	0.52	20	72	120	30	50	31	137	81

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
2489	Toluene	5	ug/kg	0.27	20	71	130	24	50	46	147	24
2515	1,2,4-Trichlorobenzene	5	ug/kg	0.27	20	50	150	20	50	50	150	20
2518	1,1,1-Trichloroethane	5	ug/kg	0.56	20	67	123	30	50	48	132	57
2522	1,1,2-Trichloroethane	5	ug/kg	0.39	20	82	116	30	50	58	128	52
2525	Trichloroethene	5	ug/kg	0.42	20	70	131	23	50	46	143	23
1428	Trichlorofluoromethane	5	ug/kg	0.34	20	50	150	20	50	50	150	20
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	5	ug/kg	1.3	20	70	130	30	50	70	130	30
2613	Vinyl chloride	5	ug/kg	0.39	20	24	152	30	50	30	136	80
2627	Xylenes (total)	10	ug/kg	0.67	60	80	114	30	150	33	135	78
337	4-Bromofluorobenzene				50	47	158	0	50	47	158	0
2735	1,2-Dichloroethane-d4				50	61	130	0	50	61	130	0
2740	Toluene-d8				50	60	143	0	50	60	143	0
2863	Dibromofluoromethane				50	59	138	0	50	59	138	0
Semi-Volatile Organics - Water Samples												
1	Acenaphthene	0.2	ug/L	0.054	50	40	110	30	50	36	110	30
5	Acenaphthylene	0.2	ug/L	0.054	50	43	110	30	50	39	110	30
24	Acetophenone	1	ug/L	0.55	50	50	130	30	50	50	130	30
122	Anthracene	0.2	ug/L	0.054	50	54	114	30	50	46	110	30
158	Atrazine	1	ug/L	0.65	50	50	130	30	50	50	130	30
3398	Benzaldehyde	1	ug/L	0.75	50	10	130	30	50	10	130	30
202	Benzo(a)anthracene	0.2	ug/L	0.052	50	55	115	30	50	52	110	30
205	Benzo(b)fluoranthene	0.2	ug/L	0.049	50	43	122	30	50	33	114	30
208	Benzo(k)fluoranthene	0.2	ug/L	0.049	50	43	124	30	50	32	121	30
210	Benzo(ghi)perylene	0.2	ug/L	0.053	50	45	120	30	50	34	116	30
211	Benzo(a)pyrene	0.2	ug/L	0.048	50	43	116	30	50	33	110	30
3474	1,1'-Biphenyl	1	ug/L	0.55	50	50	130	30	50	50	130	30
289	bis(2-Chloroethoxy)methane	1	ug/L	0.49	50	39	110	30	50	35	110	30
293	bis(2-Chloroethyl) ether	1	ug/L	0.088	50	34	113	30	50	27	110	30
302	bis(2-Ethylhexyl) phthalate	2	ug/L	0.88	50	36	163	30	50	40	140	30
348	4-Bromophenyl phenyl ether	2	ug/L	0.52	50	51	114	30	50	42	113	30
403	Butyl benzyl phthalate	1	ug/L	0.51	50	53	126	30	50	51	121	30
5101	Caprolactam	5	ug/L	0.61	50	50	130	30	50	50	130	30
2751	Carbazole	1	ug/L	0.54	50	53	120	30	50	49	114	30
518	4-Chloroaniline	2	ug/L	0.56	50	10	110	30	50	10	110	30
578	4-Chloro-3-methylphenol	2	ug/L	0.41	50	39	110	30	50	33	110	30
589	2-Chloronaphthalene	1	ug/L	0.62	50	39	110	30	50	34	110	30
600	2-Chlorophenol	1	ug/L	0.039	50	27	110	30	50	26	110	30
602	4-Chlorophenyl phenyl ether	2	ug/L	0.55	50	50	115	30	50	43	113	30
633	Chrysene	0.2	ug/L	0.048	50	55	115	30	50	52	111	30
860	Dibenz(a,h)anthracene	0.2	ug/L	0.039	50	46	122	30	50	35	118	30
863	Dibenzofuran	1	ug/L	0.54	50	46	111	30	50	41	110	30
891	Di-n-butyl phthalate	1	ug/L	0.61	50	55	122	30	50	50	117	30
918	3,3'-Dichlorobenzidine	5	ug/L	0.48	50	19	110	30	50	10	110	30
971	2,4-Dichlorophenol	2	ug/L	1.1	50	33	110	30	50	30	110	30
1082	Diethyl phthalate	1	ug/L	0.63	50	33	134	30	50	33	130	30
1145	2,4-Dimethylphenol	2	ug/L	0.56	50	12	110	30	50	11	110	30

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
1149	Dimethyl phthalate	1	ug/L	0.44	50	15	143	30	50	36	124	30
1167	4,6-Dinitro-2-methylphenol	5	ug/L	0.27	50	28	112	30	50	25	110	30
1187	2,4-Dinitrophenol	5	ug/L	3.5	50	17	112	30	50	11	119	30
1191	2,4-Dinitrotoluene	5	ug/L	0.4	50	52	123	30	50	46	119	30
1193	2,6-Dinitrotoluene	5	ug/L	0.47	50	52	119	30	50	48	115	30
1162	Di-n-octyl phthalate	1	ug/L	0.39	50	44	128	30	50	36	124	30
1414	Fluoranthene	0.2	ug/L	0.036	50	54	122	30	50	53	111	30
1417	Fluorene	0.2	ug/L	0.043	50	47	112	30	50	43	110	30
1482	Hexachlorobenzene	0.2	ug/L	0.065	50	51	112	30	50	40	113	30
1489	Hexachlorobutadiene	1	ug/L	0.51	50	13	110	30	50	14	110	30
1492	Hexachlorocyclopentadiene	10	ug/L	0.74	50	10	110	30	50	10	110	30
1497	Hexachloroethane	1	ug/L	0.58	50	12	110	30	50	10	110	30
1535	Indeno(1,2,3-cd)pyrene	0.2	ug/L	0.065	50	46	121	30	50	36	116	30
1566	Isophorone	1	ug/L	0.5	50	44	128	30	50	34	125	30
1829	2-Methylnaphthalene	0.2	ug/L	0.061	50	35	110	30	50	35	110	30
1851	2-Methylphenol	1	ug/L	0.56	50	30	110	30	50	26	110	30
1857	4-Methylphenol	1	ug/L	0.64	50	32	110	30	50	25	110	30
1932	Naphthalene	0.2	ug/L	0.069	50	31	110	30	50	32	110	30
1960	2-Nitroaniline	2	ug/L	0.43	50	43	130	30	50	31	129	30
1964	3-Nitroaniline	2	ug/L	0.67	50	45	116	30	50	23	112	30
1968	4-Nitroaniline	2	ug/L	0.47	50	45	120	30	50	26	115	30
1972	Nitrobenzene	1	ug/L	0.053	50	37	115	30	50	26	118	30
1998	2-Nitrophenol	2	ug/L	1.3	50	29	110	30	50	30	110	30
2001	4-Nitrophenol	5	ug/L	0.63	50	12	130	30	50	13	127	30
2028	N-Nitrosodiphenylamine	1	ug/L	0.46	50	53	113	30	50	28	118	30
2024	N-Nitrosodi-n-propylamine	1	ug/L	0.53	50	37	121	30	50	25	119	30
3597	2,2'-oxybis(1-Chloropropane)	1	ug/L	0.52	50	25	128	30	50	13	124	30
2118	Pentachlorophenol	5	ug/L	0.48	50	26	110	30	50	23	110	30
2154	Phenanthrene	0.2	ug/L	0.087	50	52	114	30	50	47	110	30
2155	Phenol	1	ug/L	0.96	50	14	112	30	50	16	110	30
2252	Pyrene	0.2	ug/L	0.048	50	55	120	30	50	54	115	30
2555	2,4,5-Trichlorophenol	5	ug/L	0.96	50	39	110	30	50	36	110	30
2559	2,4,6-Trichlorophenol	5	ug/L	1.4	50	35	110	30	50	34	110	30
1425	2-Fluorobiphenyl				50	28	110	0	50	28	110	0
1426	2-Fluorophenol				75	10	110	0	75	10	110	0
2512	2,4,6-Tribromophenol				75	22	120	0	75	22	120	0
2736	Nitrobenzene-d5				50	27	111	0	50	27	111	0
2737	Phenol-d5				75	10	110	0	75	10	110	0
2738	Terphenyl-d14				50	37	119	0	50	37	119	0

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
Semi-Volatile Organics - Soil/Solid Samples												
1	Acenaphthene	6.67	ug/kg	1.3	1700	46	110	30	1700	10	200	30
5	Acenaphthylene	6.67	ug/kg	1.2	1700	47	110	30	1700	10	200	30
24	Acetophenone	100	ug/kg	26	1700	50	130	30	1700	50	130	30
122	Anthracene	6.67	ug/kg	1.3	1700	56	111	30	1700	10	200	30
158	Atrazine	200	ug/kg	21	1700	50	130	30	1700	50	130	30
3398	Benzaldehyde	100	ug/kg	21	1700	10	130	30	1700	10	130	30
202	Benzo(a)anthracene	6.67	ug/kg	0.95	1700	58	111	30	1700	10	200	30
205	Benzo(b)fluoranthene	6.67	ug/kg	1.2	1700	43	124	30	1700	10	200	30
208	Benzo(k)fluoranthene	6.67	ug/kg	1.7	1700	38	122	30	1700	10	200	30
210	Benzo(ghi)perylene	6.67	ug/kg	1.3	1700	44	120	30	1700	10	200	30
211	Benzo(a)pyrene	6.67	ug/kg	1.3	1700	44	115	30	1700	10	200	30
3474	1,1'-Biphenyl	50	ug/kg	23	1700	50	130	30	1700	50	130	30
289	bis(2-Chloroethoxy)methane	100	ug/kg	22	1700	42	110	30	1700	36	110	30
293	bis(2-Chloroethyl) ether	100	ug/kg	2	1700	41	110	30	1700	32	118	30
302	bis(2-Ethylhexyl) phthalate	50	ug/kg	18	1700	56	123	30	1700	10	200	30
348	4-Bromophenyl phenyl ether	50	ug/kg	21	1700	53	112	30	1700	44	120	30
403	Butyl benzyl phthalate	50	ug/kg	19	1700	57	121	30	1700	43	138	30
5101	Caprolactam	330	ug/kg	37	1700	50	130	30	1700	50	130	30
2751	Carbazole	50	ug/kg	19	1700	56	115	30	1700	10	162	30
518	4-Chloroaniline	150	ug/kg	17	1700	25	110	30	1700	11	110	30
578	4-Chloro-3-methylphenol	150	ug/kg	21	1700	42	110	30	1700	32	117	30
589	2-Chloronaphthalene	50	ug/kg	22	1700	46	110	30	1700	40	110	30
600	2-Chlorophenol	50	ug/kg	26	1700	39	110	30	1700	32	110	30
602	4-Chlorophenyl phenyl ether	50	ug/kg	24	1700	53	110	30	1700	47	116	30
633	Chrysene	6.67	ug/kg	0.9	1700	56	111	30	1700	10	200	30
860	Dibenz(a,h)anthracene	6.67	ug/kg	1.3	1700	45	122	30	1700	10	200	30
863	Dibenzofuran	50	ug/kg	20	1700	50	110	30	1700	10	200	30
891	Di-n-butyl phthalate	50	ug/kg	19	1700	57	119	30	1700	31	145	30
918	3,3'-Dichlorobenzidine	100	ug/kg	18	1700	31	110	30	1700	10	110	30
971	2,4-Dichlorophenol	150	ug/kg	20	1700	40	110	30	1700	33	110	30
1082	Diethyl phthalate	50	ug/kg	19	1700	55	114	30	1700	48	118	30
1145	2,4-Dimethylphenol	150	ug/kg	20	1700	28	110	30	1700	19	114	30
1149	Dimethyl phthalate	50	ug/kg	21	1700	54	112	30	1700	47	116	30
1167	4,6-Dinitro-2-methylphenol	150	ug/kg	13	1700	21	110	30	1700	10	110	30
1187	2,4-Dinitrophenol	330	ug/kg	83	1700	10	110	30	1700	10	110	30
1191	2,4-Dinitrotoluene	200	ug/kg	18	1700	55	116	30	1700	42	118	30
1193	2,6-Dinitrotoluene	200	ug/kg	21	1700	54	115	30	1700	28	137	30
1162	Di-n-octyl phthalate	50	ug/kg	18	1700	45	123	30	1700	10	182	30
1414	Fluoranthene	6.67	ug/kg	1.2	1700	55	118	30	1700	10	200	30
1417	Fluorene	6.67	ug/kg	1.2	1700	51	110	30	1700	10	187	30
1482	Hexachlorobenzene	6.67	ug/kg	2.1	1700	51	110	30	1700	37	122	30
1489	Hexachlorobutadiene	50	ug/kg	26	1700	39	110	30	1700	30	110	30
1492	Hexachlorocyclopentadiene	330	ug/kg	16	1700	10	110	30	1700	10	110	30
1497	Hexachloroethane	50	ug/kg	28	1700	38	110	30	1700	13	110	30
1535	Indeno(1,2,3-cd)pyrene	6.67	ug/kg	1.5	1700	45	121	30	1700	10	200	30
1566	Isophorone	50	ug/kg	21	1700	46	117	30	1700	32	129	30

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
1829	2-Methylnaphthalene	6.67	ug/kg	1.5	1700	46	110	30	1700	10	200	30
1851	2-Methylphenol	200	ug/kg	28	1700	36	110	30	1700	19	124	30
1857	4-Methylphenol	200	ug/kg	22	1700	40	110	30	1700	27	116	30
1932	Naphthalene	6.67	ug/kg	1.6	1700	42	110	30	1700	10	200	30
1960	2-Nitroaniline	200	ug/kg	22	1700	47	124	30	1700	31	141	30
1964	3-Nitroaniline	200	ug/kg	16	1700	44	110	30	1700	24	110	30
1968	4-Nitroaniline	200	ug/kg	26	1700	50	110	30	1700	23	124	30
1972	Nitrobenzene	100	ug/kg	2.2	1700	40	110	30	1700	33	111	30
1998	2-Nitrophenol	50	ug/kg	19	1700	35	110	30	1700	17	110	30
2001	4-Nitrophenol	330	ug/kg	110	1700	24	117	30	1700	10	125	30
2028	N-Nitrosodiphenylamine	50	ug/kg	21	1700	54	112	30	1700	10	169	30
2024	N-Nitrosodi-n-propylamine	50	ug/kg	23	1700	40	114	30	1700	30	121	30
3597	2,2'-oxybis(1-Chloropropane)	100	ug/kg	26	1700	36	116	30	1700	25	124	30
2118	Pentachlorophenol	150	ug/kg	82	1700	10	110	30	1700	10	182	30
2154	Phenanthrene	6.67	ug/kg	2	1700	54	110	30	1700	10	200	30
2155	Phenol	50	ug/kg	25	1700	39	110	30	1700	10	144	30
2252	Pyrene	6.67	ug/kg	1.1	1700	58	113	30	1700	10	200	30
2555	2,4,5-Trichlorophenol	150	ug/kg	25	1700	42	110	30	1700	32	112	30
2559	2,4,6-Trichlorophenol	150	ug/kg	21	1700	37	110	30	1700	22	110	30
1425	2-Fluorobiphenyl				1700	34	110	0	1700	34	110	0
1426	2-Fluorophenol				2500	26	110	0	2500	26	110	0
2512	2,4,6-Tribromophenol				2500	10	118	0	2500	10	118	0
2736	Nitrobenzene-d5				1700	24	112	0	1700	24	112	0
2737	Phenol-d5				2500	28	110	0	2500	28	110	0
2738	Terphenyl-d14				1700	41	119	0	1700	41	119	0
Ploychlorinated Biphenyls - Water Samples												
2082	Aroclor 1016	0.2	ug/L	0.044	10	44	119	30	10	10	166	30
2085	Aroclor 1221	0.2	ug/L	0.045		0	0	0		0	0	0
2088	Aroclor 1232	0.2	ug/L	0.073		0	0	0		0	0	0
2091	Aroclor 1242	0.2	ug/L	0.06		0	0	0		0	0	0
2094	Aroclor 1248	0.2	ug/L	0.061		0	0	0		0	0	0
2097	Aroclor 1254	0.2	ug/L	0.032		0	0	0		0	0	0
2100	Aroclor 1260	0.2	ug/L	0.038	10	41	118	30	10	21	140	30
2732	Decachlorobiphenyl				0.2	10	127	0	0.2	10	127	0
2739	Tetrachloro-m-xylene				0.2	27	130	0	0.2	27	130	0
Ploychlorinated Biphenyls - Soil/Solid Samples												
2082	Aroclor 1016	33	ug/kg	21	333	34	127	30	333	10	199	30
2085	Aroclor 1221	33	ug/kg	16		0	0	0		0	0	0
2088	Aroclor 1232	33	ug/kg	14		0	0	0		0	0	0
2091	Aroclor 1242	33	ug/kg	13		0	0	0		0	0	0
2094	Aroclor 1248	33	ug/kg	17		0	0	0		0	0	0
2097	Aroclor 1254	33	ug/kg	17		0	0	0		0	0	0
2100	Aroclor 1260	33	ug/kg	17	333	32	141	30	333	10	199	30
2732	Decachlorobiphenyl				6.67	10	199	0	6.67	10	199	0
2739	Tetrachloro-m-xylene				6.67	10	196	0	6.67	10	196	0

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
#	Compound	RL	Units	MDL	Units	LCS/D LCL	LCS/D UCL	LCS/D RPD	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD	
Chlorinated Pesticides - Water Samples												
60	Aldrin	0.05	ug/L	0.0082	ug/L	54	131	30	21	159	30	
226	alpha-BHC	0.05	ug/L	0.007	ug/L	50	145	30	17	180	30	
228	beta-BHC	0.05	ug/L	0.0084	ug/L	50	147	30	21	182	30	
230	delta-BHC	0.05	ug/L	0.0087	ug/L	51	157	30	32	183	30	
232	gamma-BHC (Lindane)	0.05	ug/L	0.0064	ug/L	51	145	30	23	177	30	
497	alpha-Chlordane	0.05	ug/L	0.014	ug/L	51	137	30	24	165	30	
499	gamma-Chlordane	0.05	ug/L	0.012	ug/L	53	142	30	22	174	30	
770	4,4'-DDD	0.05	ug/L	0.0096	ug/L	44	163	30	10	199	30	
777	4,4'-DDE	0.05	ug/L	0.0097	ug/L	50	137	30	10	180	30	
780	4,4'-DDT	0.05	ug/L	0.016	ug/L	43	148	30	10	185	30	
1052	Dieldrin	0.05	ug/L	0.0075	ug/L	54	139	30	33	167	30	
1236	Endosulfan I	0.05	ug/L	0.013	ug/L	10	112	30	10	122	30	
1239	Endosulfan II	0.05	ug/L	0.012	ug/L	10	114	30	10	120	30	
1241	Endosulfan sulfate	0.05	ug/L	0.011	ug/L	50	142	30	42	165	30	
1270	Endrin	0.05	ug/L	0.011	ug/L	49	145	30	18	189	30	
1277	Endrin aldehyde	0.05	ug/L	0.011	ug/L	50	140	30	31	169	30	
1279	Endrin ketone	0.05	ug/L	0.0078	ug/L	48	141	30	10	199	30	
1470	Heptachlor	0.05	ug/L	0.008	ug/L	47	141	30	10	199	30	
1479	Heptachlor epoxide	0.05	ug/L	0.0071	ug/L	49	140	30	40	157	30	
1741	Methoxychlor	0.1	ug/L	0.032	ug/L	46	147	30	26	177	30	
2499	Toxaphene	2	ug/L	0.32	ug/L	0	0	0	0	0	0	
2732	Decachlorobiphenyl					10	139	0	10	139	0	
2739	Tetrachloro-m-xylene					25	142	0	25	142	0	
#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD	
Chlorinated Pesticides - Soil/Solid Samples												
60	Aldrin	1.7	ug/kg	1.2	ug/kg	25	149	30	10	199	30	
226	alpha-BHC	1.7	ug/kg	0.73	ug/kg	19	155	30	10	182	30	
228	beta-BHC	1.7	ug/kg	1.1	ug/kg	18	160	30	10	199	30	
230	delta-BHC	1.7	ug/kg	1.2	ug/kg	17	169	30	10	199	30	
232	gamma-BHC (Lindane)	1.7	ug/kg	0.74	ug/kg	21	155	30	10	199	30	
497	alpha-Chlordane	1.7	ug/kg	0.94	ug/kg	22	151	30	10	199	30	
499	gamma-Chlordane	1.7	ug/kg	0.42	ug/kg	21	160	30	10	199	30	
770	4,4'-DDD	1.7	ug/kg	0.62	ug/kg	15	176	30	10	199	30	
777	4,4'-DDE	1.7	ug/kg	0.39	ug/kg	23	152	30	10	199	30	
780	4,4'-DDT	1.7	ug/kg	0.63	ug/kg	13	152	30	10	199	30	
1052	Dieldrin	1.7	ug/kg	0.47	ug/kg	24	152	30	10	199	30	
1236	Endosulfan I	1.7	ug/kg	0.52	ug/kg	10	112	30	10	199	30	
1239	Endosulfan II	1.7	ug/kg	0.82	ug/kg	10	113	30	10	127	30	
1241	Endosulfan sulfate	1.7	ug/kg	0.87	ug/kg	16	154	30	10	167	30	
1270	Endrin	1.7	ug/kg	0.5	ug/kg	21	156	30	10	199	30	
1277	Endrin aldehyde	1.7	ug/kg	1	ug/kg	17	156	30	10	192	30	
1279	Endrin ketone	1.7	ug/kg	0.63	ug/kg	22	147	30	10	199	30	
1470	Heptachlor	1.7	ug/kg	1.1	ug/kg	24	147	30	10	199	30	

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
1479	Heptachlor epoxide	1.7	ug/kg	0.8	ug/kg	21	151	30	10	199	30	
1741	Methoxychlor	3.3	ug/kg	1.5	ug/kg	14	160	30	10	199	30	
2499	Toxaphene	67	ug/kg	19	ug/kg	0	0	0	0	0	0	
2732	Decachlorobiphenyl					10	199	0	10	199	0	
2739	Tetrachloro-m-xylene					10	199	0	10	199	0	

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
Herbicides - Water Samples											
690	2,4-D	4	ug/L	1.5	ug/L	35	110	30	26	113	30
2291	2,4,5-TP (Silvex)	1	ug/L	0.16	ug/L	41	110	30	34	112	30
2924	2,4-Dichlorophenylacetic acid					32	112	0	32	112	0

#	Compound	RL	Units	MDL	Units	LCL	UCL	RPD	LCL	UCL	RPD
Herbicides - Soil/Solid Samples											
690	2,4-D	80	ug/kg	36	ug/kg	33	110	30	15	110	30
2291	2,4,5-TP (Silvex)	20	ug/kg	2.2	ug/kg	42	110	30	10	117	30
2924	2,4-Dichlorophenylacetic acid					19	122	0	19	122	0

#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
1701	Mercury	0.2	ug/L	0.12	5	81	123	20	1	69	134	20

#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
1701	Mercury	0.1	mg/kg	0.015	0.833333	73	121	20	0.166667	11	192	20

6010 Metals

#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
88	Aluminum	20	mg/kg	9.6	200	80	120	20	200	75	125	20
128	Antimony	6	mg/kg	0.39	50	80	120	20	50	75	125	20
140	Arsenic	30	mg/kg	0.3	200	80	120	20	200	75	125	20
194	Barium	20	mg/kg	0.071	200	80	120	20	200	75	125	20
222	Beryllium	0.5	mg/kg	0.043	5	80	120	20	5	75	125	20
313	Boron	20	mg/kg	3.4	100	80	120	20	100	75	125	20
411	Cadmium	0.5	mg/kg	0.036	5	80	120	20	5	75	125	20
413	Calcium	500	mg/kg	16	5000	80	120	20	5000	75	125	20
2952	Chromium	1	mg/kg	0.2	20	80	120	20	20	75	125	20
637	Cobalt	5	mg/kg	0.16	50	80	120	20	50	75	125	20
643	Copper	2.5	mg/kg	0.74	25	80	120	20	25	75	125	20
1539	Iron	10	mg/kg	4.9	100	73	137	20	100	75	125	20
1605	Lead	10	mg/kg	0.19	50	80	120	20	50	75	125	20
1618	Magnesium	500	mg/kg	5.1	5000	80	120	20	5000	75	125	20
1659	Manganese	1.5	mg/kg	0.074	50	80	120	20	50	75	125	20
1906	Molybdenum	4	mg/kg	0.27	100	80	120	20	100	75	125	20
1956	Nickel	4	mg/kg	0.27	50	80	120	20	50	75	125	20
2214	Potassium	500	mg/kg	6.2	5000	80	120	20	5000	75	125	20
2281	Selenium	25	mg/kg	0.45	200	80	120	20	200	75	125	20
2285	Silver	1	mg/kg	0.1	5	80	120	20	5	75	125	20

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
2315	Sodium	500	mg/kg	66	5000	80	120	20	5000	75	125	20
2477	Thallium	200	mg/kg	0.55	200	80	120	20	200	75	125	20
2479	Tin	10	mg/kg	0.5	200	80	120	20	200	75	125	20
2482	Titanium	5	mg/kg	0.57	100	80	120	20	100	75	125	20
2607	Vanadium	5	mg/kg	0.12	50	80	120	20	50	75	125	20
2649	Zinc	2	mg/kg	1	50	80	120	20	50	75	125	20

#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
88	Aluminum	200	ug/L	97	2000	80	120	20	2000	75	125	20
128	Antimony	60	ug/L	1.8	500	80	120	20	500	75	125	20
140	Arsenic	300	ug/L	3.2	2000	80	120	20	2000	75	125	20
194	Barium	200	ug/L	0.67	2000	80	120	20	2000	75	125	20
222	Beryllium	5	ug/L	0.46	50	80	120	20	50	75	125	20
313	Boron	200	ug/L	34	1000	80	120	20	1000	75	125	20
411	Cadmium	5	ug/L	0.66	50	80	120	20	50	75	125	20
413	Calcium	5000	ug/L	130	50000	80	120	20	50000	75	125	20
2952	Chromium	10	ug/L	2.2	200	80	120	20	200	75	125	20
637	Cobalt	50	ug/L	1.7	500	80	120	20	500	75	125	20
643	Copper	25	ug/L	4.5	250	80	120	20	250	75	125	20
1539	Iron	100	ug/L	81	1000	77	122	20	1000	75	125	20
1605	Lead	100	ug/L	1.9	500	80	120	20	500	75	125	20
1618	Magnesium	5000	ug/L	34	50000	80	120	20	50000	75	125	20
1659	Manganese	15	ug/L	0.41	500	80	120	20	500	75	125	20
1906	Molybdenum	40	ug/L	1.3	1000	80	120	20	1000	75	125	20
1956	Nickel	40	ug/L	3.2	500	80	120	20	500	75	125	20
2214	Potassium	5000	ug/L	72	50000	80	120	20	50000	75	125	20
2281	Selenium	250	ug/L	4.1	2000	80	120	20	2000	75	125	20
2285	Silver	10	ug/L	2.2	50	80	120	20	50	75	125	20
2315	Sodium	5000	ug/L	590	50000	80	120	20	50000	75	125	20
2477	Thallium	200	ug/L	4.7	2000	80	120	20	2000	75	125	20
2479	Tin	100	ug/L	3.3	2000	80	120	20	2000	75	125	20
2482	Titanium	50	ug/L	5.3	1000	80	120	20	1000	75	125	20
2607	Vanadium	50	ug/L	0.64	500	80	120	20	500	75	125	20
2649	Zinc	20	ug/L	5	500	80	120	20	500	75	125	20

6020 Metals

#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
88	Aluminum	50	ug/L	19	1000	77	123	20	1000	63	128	20
128	Antimony	2	ug/L	0.13	100	57	110	20	100	44	153	20
140	Arsenic	5	ug/L	0.4	100	86	118	20	100	82	123	20
194	Barium	1	ug/L	0.19	100	83	110	20	100	45	144	20
222	Beryllium	1	ug/L	0.2	100	84	120	20	100	77	124	20
313	Boron	20	ug/L	3.9	100	80	120	20	100	80	120	20
411	Cadmium	1	ug/L	0.13	100	89	114	20	100	78	117	20
413	Calcium	1000	ug/L	22	1000	80	120	20	1000	70	130	20
2952	Chromium	2	ug/L	0.71	100	81	110	20	100	72	110	20

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
637	Cobalt	1	ug/L	0.058	100	82	113	20	100	67	114	20
643	Copper	2	ug/L	0.29	100	82	113	20	100	60	123	20
1539	Iron	50	ug/L	26	1000	80	118	20	1000	22	169	20
1605	Lead	1	ug/L	0.18	100	84	113	20	100	73	115	20
1618	Magnesium	1000	ug/L	17	1000	80	120	20	1000	70	130	20
1659	Manganese	1	ug/L	0.83	100	78	111	20	100	10	172	20
1906	Molybdenum	2	ug/L	0.093	100	62	111	20	100	80	110	20
1956	Nickel	2	ug/L	0.2	100	80	111	20	100	72	111	20
2214	Potassium	1000	ug/L	8.3	1000	80	120	20	1000	70	130	20
2281	Selenium	5	ug/L	1.2	100	90	128	20	100	72	148	20
2285	Silver	1	ug/L	0.08	100	83	111	20	100	10	139	20
2315	Sodium	1000	ug/L	6.9	1000	0	0	0	1000	0	0	0
2353	Strontium	10	ug/L	0.33	100	82	110	20	100	20	192	20
2477	Thallium	1	ug/L	0.14	100	82	113	20	100	69	117	20
2479	Tin	10	ug/L	0.31	100	77	110	20	100	50	117	20
2602	Tungsten	10	ug/L	0.37	100	78	110	20	100	43	133	20
2607	Vanadium	20	ug/L	0.44	100	82	110	20	100	70	112	20
2649	Zinc	20	ug/L	2.3	100	90	129	20	100	49	156	20

#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
88	Aluminum	5	mg/kg	1.3	100	80	120	20	100	70	130	20
128	Antimony	0.2	mg/kg	0.024	10	68	113	20	10	75	125	20
140	Arsenic	0.5	mg/kg	0.062	10	73	110	20	10	23	131	20
194	Barium	0.1	mg/kg	0.046	10	70	110	20	10	10	199	20
222	Beryllium	0.1	mg/kg	0.047	10	79	110	20	10	58	112	20
313	Boron	2	mg/kg	0.18	10	80	120	20	10	80	120	20
411	Cadmium	0.1	mg/kg	0.0078	10	74	110	20	10	58	110	20
2952	Chromium	0.2	mg/kg	0.04	10	70	110	20	10	10	199	20
637	Cobalt	0.1	mg/kg	0.0038	10	74	110	20	10	55	110	20
643	Copper	0.2	mg/kg	0.043	10	73	110	20	10	10	199	20
1539	Iron	5	mg/kg	1	100	80	120	20	100	70	130	20
1605	Lead	0.1	mg/kg	0.013	10	75	110	20	10	10	199	20
1618	Magnesium	100	mg/kg	2.1	100	80	120	20	100	70	130	20
1659	Manganese	0.1	mg/kg	0.047	10	80	120	20	10	10	199	20
1906	Molybdenum	0.2	mg/kg	0.063	10	30	140	20	10	10	199	20
1956	Nickel	0.1	mg/kg	0.021	10	75	110	20	10	10	176	20
2214	Potassium	100	mg/kg	1.1	100	80	120	20	100	70	130	20
2281	Selenium	0.5	mg/kg	0.09	10	65	97	20	10	39	116	20
2285	Silver	0.1	mg/kg	0.016	10	60	114	20	10	75	125	20
2315	Sodium	100	mg/kg	2.4	100	80	120	20	100	70	130	20
2353	Strontium	1	mg/kg	0.063	10	80	120	20	10	52	110	20
2477	Thallium	0.1	mg/kg	0.013	10	71	110	20	10	62	110	20
2479	Tin	1	mg/kg	0.094	10	80	120	20	10	75	125	20
2607	Vanadium	0.5	mg/kg	0.041	10	72	110	20	10	39	129	20
2649	Zinc	1	mg/kg	0.44	10	72	113	20	10	10	199	20

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
Inorganics												
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
667	Total Cyanide	0.5	mg/kg	0.1		68	123	20	2	50	134	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
667	Total Cyanide	0.01	mg/L	0.005		69	118	20	0.04	42	140	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
3041	Total Alkalinity	5	mg/L	1.9		90	127	20	500	10	160	24
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
512	Chloride	1	mg/L	0.1	50	90	110	20	50	80	120	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
3655	Dissolved Organic Carbon	1	mg/L	0.24		88	115	20	25	72	136	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
1469	Hardness, as CaCO3	33.075	mg/L	33.075		88	110	20	500	87	114	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
3744	Nitrate as N	0.1	mg/L	0.023	2.5	90	110	20	2.5	80	120	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
3746	Nitrite as N	0.1	mg/L	0.012	2.5	90	110	20	2.5	80	120	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
2363	Sulfate	1	mg/L	0.12	50	90	110	20	50	80	120	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
3693	Acid-soluble sulfide	1	mg/L	0.86	20	75	125	20	20	75	125	20
3693	Acid-soluble sulfide	100	mg/kg	11	20	75	125	20	20	75	125	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
3377	Ethane	0.5	ug/L	0.27		74	138	30		74	138	30
3386	Ethene	0.5	ug/L	0.24		73	140	30		73	140	30
2841	Methane	0.5	ug/L	0.17		75	127	30		75	127	30

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
TCLP/Waster Characterization												
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
196	Benzene	0.025	mg/L	0.13	50	76	118	30	50	76	117	30
3271	2-Butanone (MEK)	0.25	mg/L	0.57		0	0	0		0	0	0
463	Carbon tetrachloride	0.025	mg/L	0.13	50	71	124	30	50	72	124	30
521	Chlorobenzene	0.025	mg/L	0.15	50	76	113	30	50	72	114	30
569	Chloroform	0.025	mg/L	0.16	50	82	117	30	50	82	117	30
936	1,2-Dichloroethane	0.025	mg/L	0.22	50	78	122	30	50	80	120	30
946	1,1-Dichloroethylene	0.07	mg/L	0.19		0	0	0		0	0	0
2446	Tetrachloroethylene	0.07	mg/L	0.29		0	0	0		0	0	0
2526	Trichloroethylene	0.05	mg/L	0.17		0	0	0		0	0	0
2613	Vinyl chloride	0.025	mg/L	0.22	50	47	123	30	50	54	118	30
337	4-Bromofluorobenzene				50	84	125	0	50	84	125	0
2735	1,2-Dichloroethane-d4				50	80	122	0	50	80	122	0
2740	Toluene-d8				50	90	122	0	50	90	122	0
2863	Dibromofluoromethane				50	86	124	0	50	86	125	0
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
2778	m-Cresol & p-Cresol	0.04	mg/L	0.75	0.4	27	110	30	0.4	46	109	32
910	1,4-Dichlorobenzene	0.004	mg/L	0.52	0.2	16	110	30	0.2	18	110	36
1191	2,4-Dinitrotoluene	0.02	mg/L	0.4	0.2	45	126	30	0.2	31	131	32
1482	Hexachlorobenzene	0.02	mg/L	0.065	0.2	47	116	30	0.2	36	132	22
1489	Hexachlorobutadiene	0.02	mg/L	0.51	0.2	10	110	30	0.2	18	116	32
1497	Hexachloroethane	0.02	mg/L	0.58	0.2	10	110	30	0.2	18	110	33
1853	o-Cresol	0.004	mg/L	0.56	0.2	24	110	30	0.2	33	115	31
1972	Nitrobenzene	0.004	mg/L	0.053	0.2	35	117	30	0.2	19	211	59
2118	Pentachlorophenol	0.04	mg/L	0.48	0.2	12	110	30	0.2	10	140	56
2256	Pyridine	0.02	mg/L	0.78	0.2	10	110	30	0.2	10	148	65
2555	2,4,5-Trichlorophenol	0.02	mg/L	0.96	0.2	35	111	30	0.2	24	143	22
2559	2,4,6-Trichlorophenol	0.02	mg/L	1.4	0.2	32	110	30	0.2	36	135	27
1425	2-Fluorobiphenyl					22	110	0		22	110	0
1426	2-Fluorophenol					10	110	0		10	110	0
2512	2,4,6-Tribromophenol					17	117	0		17	117	0
2736	Nitrobenzene-d5					29	111	0		29	111	0
2737	Phenol-d5					10	110	0		10	110	0
2738	Terphenyl-d14					40	119	0		40	119	0
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
233	Lindane	0.0005	mg/L	0.0064		56	110	88		50	150	50
476	Chlordane (technical)	0.005	mg/L	0.033		0	0	0		0	0	0
1270	Endrin	0.0005	mg/L	0.011		50	110	93		50	150	50
1470	Heptachlor	0.0005	mg/L	0.008		57	110	88		50	150	50
1479	Heptachlor epoxide	0.0005	mg/L	0.0071		56	110	86		50	150	50
1741	Methoxychlor	0.001	mg/L	0.032		41	126	94		50	150	50
2499	Toxaphene	0.02	mg/L	0.32		0	0	0		0	0	0
2732	Decachlorobiphenyl					31	115	0		31	115	0
2739	Tetrachloro-m-xylene					47	110	0		47	110	0

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
690	2,4-D	0.5	mg/L	1.5		35	136	50		54	114	67
2291	2,4,5-TP (Silvex)	0.1	mg/L	0.16		0	0	0		0	0	0
2924	2,4-Dichlorophenylacetic acid					37	116	0		37	116	0
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
140	Arsenic	0.5	mg/L	0.0032	2	50	150	20	5	50	150	20
194	Barium	10	mg/L	0.00067	2	50	150	20	50	50	150	20
411	Cadmium	0.1	mg/L	0.00066	0.05	50	150	20	1	50	150	20
2952	Chromium	0.5	mg/L	0.0022	0.2	50	150	20	5	50	150	20
1605	Lead	0.5	mg/L	0.0019	0.5	50	150	20	5	50	150	20
2281	Selenium	0.25	mg/L	0.0041	2	50	150	20	1	50	150	20
2285	Silver	0.5	mg/L	0.0022	0.05	50	150	20	1	50	150	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
1701	Mercury	0.002	mg/L	0.00012	5	50	150	20	5	50	150	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
2673	Flashpoint		deg F			75	125	20		75	125	20
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
2204	pH (solid)		No Units			97	103	20		97	103	20

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

# AIR Samples	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
#	Compound	RL	Units	MDL	AMT	LCL	UCL	RPD				
3036	Acetaldehyde	18	ug/m3	9		0	0	0				
11	Acetone	24	ug/m3	5.9		70	130	25				
20	Acetonitrile	34	ug/m3	17		0	0	0				
39	Acrolein	23	ug/m3	11		0	0	0				
46	Acrylonitrile	32	ug/m3	15		0	0	0				
2839	alpha-Methylstyrene	9.6	ug/m3	2.4		0	0	0				
196	Benzene	9.6	ug/m3	4.8		0	0	0				
220	Benzyl chloride	130	ug/m3	41		0	0	0				
318	Bromobenzene	64	ug/m3	19		0	0	0				
323	Bromodichloromethane	13	ug/m3	6.7		70	130	25				
333	Vinyl bromide	8.6	ug/m3	4.3		0	0	0				
340	Bromoform	21	ug/m3	5.2		0	0	0				
343	Bromomethane	16	ug/m3	7.8		70	130	25				
355	1,3-Butadiene	8.8	ug/m3	2		0	0	0				
5236	Butane	7.1	ug/m3	3.6		0	0	0				
3271	2-Butanone (MEK)	29	ug/m3	5.9		0	0	0				
1772	tert-Butyl alcohol	45	ug/m3	21		0	0	0				
393	n-Butylbenzene	11	ug/m3	3.3		0	0	0				
395	sec-Butylbenzene	11	ug/m3	5.5		0	0	0				
398	tert-Butylbenzene	11	ug/m3	5.5		0	0	0				
459	Carbon disulfide	31	ug/m3	6.2		70	130	25				
463	Carbon tetrachloride	13	ug/m3	6.3		0	0	0				
521	Chlorobenzene	9.2	ug/m3	4.6		0	0	0				
535	Dibromochloromethane	17	ug/m3	8.5		0	0	0				
2922	Chlorodifluoromethane	35	ug/m3	5.3		0	0	0				
550	Chloroethane	10	ug/m3	4		0	0	0				
569	Chloroform	9.8	ug/m3	4.9		70	130	25				
574	Chloromethane	8.2	ug/m3	2.1		0	0	0				
606	Allyl chloride	12	ug/m3	4.7		0	0	0				
614	2-Chlorotoluene	10	ug/m3	5.2		0	0	0				
669	Cyclohexane	6.9	ug/m3	3.4		0	0	0				
676	Cyclohexanone	40	ug/m3	20		0	0	0				
539	1,2-Dibromo-3-chloropropane	96	ug/m3	48		0	0	0				
3261	1,2-Dibromoethane (EDB)	15	ug/m3	7.7		0	0	0				
888	Dibromomethane	21	ug/m3	11		0	0	0				
904	1,2-Dichlorobenzene	12	ug/m3	5.4		0	0	0				
907	1,3-Dichlorobenzene	12	ug/m3	4.8		70	130	25				
910	1,4-Dichlorobenzene	36	ug/m3	18		0	0	0				
922	trans-1,4-Dichloro-2-butene	200	ug/m3	51		0	0	0				
924	Dichlorodifluoromethane	9.9	ug/m3	4.9		0	0	0				
933	1,1-Dichloroethane	8.1	ug/m3	4		0	0	0				
936	1,2-Dichloroethane	8.1	ug/m3	4		70	130	25				
948	cis-1,2-Dichloroethene	7.9	ug/m3	3.2		0	0	0				
950	trans-1,2-Dichloroethene	7.9	ug/m3	4		70	130	25				

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
943	1,1-Dichloroethene	7.9	ug/m3	4		0	0	0				
986	1,2-Dichloropropane	14	ug/m3	6.9		0	0	0				
989	1,3-Dichloropropane	46	ug/m3	4.6		0	0	0				
990	2,2-Dichloropropane	9.2	ug/m3	4.6		0	0	0				
998	cis-1,3-Dichloropropene	14	ug/m3	6.8		70	130	25				
1000	trans-1,3-Dichloropropene	9.1	ug/m3	4.5		0	0	0				
996	1,1-Dichloropropene	23	ug/m3	4.5		0	0	0				
1015	1,2-Dichloro-1,1,2,2-tetrafluoroethane	14	ug/m3	7		0	0	0				
1095	Diisobutyl ketone	58	ug/m3	29		0	0	0				
1199	1,4-Dioxane	36	ug/m3	7.2		0	0	0				
1290	Ethanol	75	ug/m3	38		0	0	0				
5441	Tert-amyl methyl ether (TAME)	8.3	ug/m3	2.1		0	0	0				
5440	Ethyl-t-Butyl Ether (ETBE)	8.3	ug/m3	1.7		0	0	0				
1325	Ethyl acetate	18	ug/m3	9		0	0	0				
1332	Ethylbenzene	8.7	ug/m3	4.3		70	130	25				
1355	Diethyl ether	6	ug/m3	3		0	0	0				
3790	4-Ethyltoluene	9.8	ug/m3	3.4		70	130	25				
2790	Freon 113	15	ug/m3	7.6		0	0	0				
1017	Freon 114	14	ug/m3	7		0	0	0				
1481	n-Heptane	12	ug/m3	6.1		0	0	0				
1489	Hexachlorobutadiene	43	ug/m3	14		0	0	0				
1514	n-Hexane	7	ug/m3	3.5		0	0	0				
1515	2-Hexanone	41	ug/m3	8.2		0	0	0				
1536	Iodomethane	29	ug/m3	12		0	0	0				
1571	Isopropanol	24	ug/m3	9.8		0	0	0				
1579	Cumene	9.8	ug/m3	4.9		0	0	0				
1578	Isopropylbenzene	9.8	ug/m3	4.9		0	0	0				
5439	Diisopropyl Ether (DIPE)	8.3	ug/m3	4.2		0	0	0				
3795	4-Isopropyltoluene (p-Cymene)	11	ug/m3	2.7		0	0	0				
1719	Methanol	33	ug/m3	6.5		0	0	0				
1811	Methylene chloride	6.9	ug/m3	2.8		0	0	0				
1823	Methyl methacrylate	29	ug/m3	14		0	0	0				
3283	4-Methyl-2-pentanone (MIBK)	41	ug/m3	8.2		0	0	0				
3794	Methyl tert-butyl ether (MTBE)	7.2	ug/m3	3.6		0	0	0				
1932	Naphthalene	31	ug/m3	16		0	0	0				
2748	n-Nonane	52	ug/m3	5.2		0	0	0				
2047	n-Octane	47	ug/m3	7		0	0	0				
2125	Pentane	5.9	ug/m3	3		0	0	0				
3440	Propane	9	ug/m3	3.6		0	0	0				
1570	2-Propanol	24	ug/m3	9.8		0	0	0				
2247	n-Propylbenzene	9.8	ug/m3	4.9		0	0	0				
3448	Propylene	8.6	ug/m3	3.4		0	0	0				
2355	Styrene	8.5	ug/m3	4.2		70	130	25				
2437	1,1,1,2-Tetrachloroethane	14	ug/m3	3.4		0	0	0				
2439	1,1,2,2-Tetrachloroethane	14	ug/m3	6.8		0	0	0				
2445	Tetrachloroethene	14	ug/m3	6.8		0	0	0				
2469	Tetrahydrofuran	15	ug/m3	2.9		0	0	0				

LABORATORY QUALITY CONTROL REFERENCE DATA
TESTAMERICA - NORTH CANTON, TESTAMERICA - LOS ANGELES (AIR)

#	Compound	RL	Units	MDL	AMT	LCS LCL	LCS UCL	LCS RPD	AMT	MS/MSD LCL	MS/MSD UCL	MS/MSD RPD
2489	Toluene	7.5	ug/m3	3.8		0	0	0				
2909	TPH (as Gasoline)	2000	ug/m3	820		0	0	0				
4920	TNMOC as Hexane	35	ug/m3	3.5		0	0	0				
2514	1,2,3-Trichlorobenzene	44	ug/m3	22		0	0	0				
2515	1,2,4-Trichlorobenzene	37	ug/m3	18		0	0	0				
2518	1,1,1-Trichloroethane	11	ug/m3	5.4		0	0	0				
2522	1,1,2-Trichloroethane	11	ug/m3	5.4		70	130	25				
2525	Trichloroethene	11	ug/m3	5.4		0	0	0				
1428	Trichlorofluoromethane	11	ug/m3	5.6		70	130	25				
2563	1,2,3-Trichloropropane	12	ug/m3	6		0	0	0				
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	15	ug/m3	7.6		70	130	25				
2587	1,2,4-Trimethylbenzene	15	ug/m3	6.4		0	0	0				
2592	1,3,5-Trimethylbenzene	15	ug/m3	5.4		0	0	0				
1564	2,2,4-Trimethylpentane	9.3	ug/m3	4.7		0	0	0				
2610	Vinyl acetate	35	ug/m3	7		0	0	0				
2613	Vinyl chloride	7.6	ug/m3	3.8		70	130	25				
2940	m-Xylene & p-Xylene	17	ug/m3	8.7		0	0	0				
2623	o-Xylene	8.7	ug/m3	4.3		0	0	0				
2627	Xylenes (total)	17	ug/m3	4.3		0	0	0				
5635	1,1-Dichloro-1-fluoroethane	9.6	ug/m3	2.4		0	0	0				
337	4-Bromofluorobenzene					70	130	0				
2735	1,2-Dichloroethane-d4					70	130	0				
2740	Toluene-d8					70	130	0				

Table: 15
Analytical Group: EPA 8290
SOP Reference: SAC-ID-0005
Matrix: Aqueous

Chemical	CAS Number	Laboratory-Specific	
		QLs (pg/L)	MDLs[1]
2,3,7,8-TCDF	51207-31-9	10	EDL
2,3,7,8-TCDD	1746-01-6	10	EDL
1,2,3,7,8-PeCDF	57117-41-6	50	EDL
2,3,4,7,8-PeCDF	57117-31-4	50	EDL
1,2,3,7,8-PeCDD	40321-76-4	50	EDL
1,2,3,4,7,8-HxCDF	70648-26-9	50	EDL
1,2,3,6,7,8-HxCDF	57117-44-9	50	EDL
2,3,4,6,7,8-HxCDF	60851-34-5	50	EDL
1,2,3,7,8,9-HxCDF	72918-21-9	50	EDL
1,2,3,4,7,8-HxCDD	39227-28-6	50	EDL
1,2,3,6,7,8-HxCDD	57653-85-7	50	EDL
1,2,3,7,8,9-HxCDD	19408-74-3	50	EDL
1,2,3,4,6,7,8-HpCDF	67562-39-4	50	EDL
1,2,3,4,7,8,9-HpCDF	55673-89-7	50	EDL
1,2,3,4,6,7,8-HpCDD	35822-46-9	50	EDL
OCDF	39001-02-0	100	EDL
OCDD	3268-87-9	100	EDL

Notes:
1 Estimated Detection Limit (EDL) - For each chemical not detected, an EDL is calculated. The sample specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, and so forth. Because of the toxicological significance of dioxins, the EDL value is reported for nondetected chemicals rather than reporting the QL.

TestAmerica West Sacramento
8290 AQ Control Limits

	LCS/LCSD Control Limits					MS/MSD Control Limits				
Compound	AMT	Units	LCL	UCL	RPD	AMT	Units	LCL	UCL	RPD
2,3,7,8-TCDF	200	pg/L	75	142	20	200	pg/L	75	142	20
Total TCDF			0	0	0			0	0	0
1,2,3,7,8-PeCDF	1000	pg/L	80	140	20	1000	pg/L	80	140	20
2,3,4,7,8-PeCDF	1000	pg/L	71	144	20	1000	pg/L	71	144	20
Total PeCDF			0	0	0			0	0	0
1,2,3,4,7,8-HxCDF	1000	pg/L	64	149	20	1000	pg/L	64	149	20
1,2,3,6,7,8-HxCDF	1000	pg/L	56	161	20	1000	pg/L	56	161	20
1,2,3,7,8,9-HxCDF	1000	pg/L	53	163	20	1000	pg/L	53	163	20
2,3,4,6,7,8-HxCDF	1000	pg/L	60	169	20	1000	pg/L	60	169	20
Total HxCDF			0	0	0			0	0	0
1,2,3,4,6,7,8-HpCDF	1000	pg/L	78	141	20	1000	pg/L	78	141	20
1,2,3,4,7,8,9-HpCDF	1000	pg/L	80	146	20	1000	pg/L	80	146	20
Total HpCDF			0	0	0			0	0	0
OCDF	2000	pg/L	76	147	20	2000	pg/L	76	147	20
2,3,7,8-TCDD	200	pg/L	71	128	20	200	pg/L	71	128	20
Total TCDD			0	0	0			0	0	0
1,2,3,7,8-PeCDD	1000	pg/L	74	139	20	1000	pg/L	74	139	20
Total PeCDD			0	0	0			0	0	0
1,2,3,7,8,9-HxCDD	1000	pg/L	60	147	20	1000	pg/L	60	147	20
1,2,3,4,7,8-HxCDD	1000	pg/L	65	144	20	1000	pg/L	65	144	20
1,2,3,6,7,8-HxCDD	1000	pg/L	73	142	20	1000	pg/L	73	142	20
Total HxCDD			0	0	0			0	0	0
1,2,3,4,6,7,8-HpCDD	1000	pg/L	79	137	20	1000	pg/L	79	137	20
Total HpCDD			0	0	0			0	0	0
OCDD	2000	pg/L	71	147	20	2000	pg/L	71	147	20

	Internal Standar Control Limits			
Compound	AMT	Units	LCL	UCL
13C-2,3,7,8-TCDF	2000	pg/L	40	135
13C-1,2,3,7,8-PeCDF	2000	pg/L	40	135
13C-1,2,3,4,7,8-HxCDF	2000	pg/L	40	135
13C-1,2,3,4,6,7,8-HpCDF	2000	pg/L	40	135
13C-2,3,7,8-TCDD	2000	pg/L	40	135
13C-1,2,3,7,8-PeCDD	2000	pg/L	40	135
13C-1,2,3,6,7,8-HxCDD	2000	pg/L	40	135
13C-1,2,3,4,6,7,8-HpCDD	2000	pg/L	40	135
13C-OCDD	4000	pg/L	40	135

SAP Worksheet: 15
Analytical Group: EPA 8290
SOP Reference: SAC-ID-0005
Matrix: Waste

Chemical	CAS Number	Laboratory-Specific QLs (ng/g)	Laboratory-Specific MDLs[1]
2,3,7,8-TCDF	51207-31-9	0.1	EDL
2,3,7,8-TCDD	1746-01-6	0.1	EDL
1,2,3,7,8-PeCDF	57117-41-6	0.5	EDL
2,3,4,7,8-PeCDF	57117-31-4	0.5	EDL
1,2,3,7,8-PeCDD	40321-76-4	0.5	EDL
1,2,3,4,7,8-HxCDF	70648-26-9	0.5	EDL
1,2,3,6,7,8-HxCDF	57117-44-9	0.5	EDL
2,3,4,6,7,8-HxCDF	60851-34-5	0.5	EDL
1,2,3,7,8,9-HxCDF	72918-21-9	0.5	EDL
1,2,3,4,7,8-HxCDD	39227-28-6	0.5	EDL
1,2,3,6,7,8-HxCDD	57653-85-7	0.5	EDL
1,2,3,7,8,9-HxCDD	19408-74-3	0.5	EDL
1,2,3,4,6,7,8-HpCDF	67562-39-4	0.5	EDL
1,2,3,4,7,8,9-HpCDF	55673-89-7	0.5	EDL
1,2,3,4,6,7,8-HpCDD	35822-46-9	0.5	EDL
OCDF	39001-02-0	1	EDL
OCDD	3268-87-9	1	EDL

Notes:
1 Estimated Detection Limit (EDL) - For each chemical not detected, an EDL is calculated. The sample specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample size, dilution, and so forth. Because of the toxicological significance of dioxins, the EDL value is reported for nondetected chemicals rather than reporting the QL.

TestAmerica West Sacramento
8290 Solid Control Limits

	LCS/LCSD Control Limits					MS/MSD Control Limits				
Compound	AMT	Units	LCL	UCL	RPD	AMT	Units	LCL	UCL	RPD
2,3,7,8-TCDF	20	pg/g	80	146	20	20	pg/g	80	146	20
Total TCDF			0	0	0			0	0	0
1,2,3,7,8-PeCDF	100	pg/g	84	143	20	100	pg/g	84	143	20
2,3,4,7,8-PeCDF	100	pg/g	76	157	20	100	pg/g	76	157	20
Total PeCDF			0	0	0			0	0	0
1,2,3,4,7,8-HxCDF	100	pg/g	78	141	20	100	pg/g	78	141	20
1,2,3,6,7,8-HxCDF	100	pg/g	78	144	20	100	pg/g	78	144	20
1,2,3,7,8,9-HxCDF	100	pg/g	70	144	20	100	pg/g	70	144	20
2,3,4,6,7,8-HxCDF	100	pg/g	73	157	20	100	pg/g	73	157	20
Total HxCDF			0	0	0			0	0	0
1,2,3,4,6,7,8-HpCDF	100	pg/g	79	143	20	100	pg/g	79	143	20
1,2,3,4,7,8,9-HpCDF	100	pg/g	79	150	20	100	pg/g	79	150	20
Total HpCDF			0	0	0			0	0	0
OCDF	200	pg/g	70	158	20	200	pg/g	70	158	20
2,3,7,8-TCDD	20	pg/g	77	133	20	20	pg/g	77	133	20
Total TCDD			0	0	0			0	0	0
1,2,3,7,8-PeCDD	100	pg/g	74	145	20	100	pg/g	74	145	20
Total PeCDD			0	0	0			0	0	0
1,2,3,7,8,9-HxCDD	100	pg/g	68	139	20	100	pg/g	68	139	20
1,2,3,4,7,8-HxCDD	100	pg/g	68	146	20	100	pg/g	68	146	20
1,2,3,6,7,8-HxCDD	100	pg/g	79	141	20	100	pg/g	79	141	20
Total HxCDD			0	0	0			0	0	0
1,2,3,4,6,7,8-HpCDD	100	pg/g	74	147	20	100	pg/g	74	147	20
Total HpCDD			0	0	0			0	0	0
OCDD	200	pg/g	75	153	20	200	pg/g	75	153	20

	Internal Standard Control Limits			
Compound	AMT	Units	LCL	UCL
13C-2,3,7,8-TCDF	200	pg/g	40	135
13C-1,2,3,7,8-PeCDF	200	pg/g	40	135
13C-1,2,3,4,7,8-HxCDF	200	pg/g	40	135
13C-1,2,3,4,6,7,8-HpCDF	200	pg/g	40	135
13C-2,3,7,8-TCDD	200	pg/g	40	135
13C-1,2,3,7,8-PeCDD	200	pg/g	40	135
13C-1,2,3,6,7,8-HxCDD	200	pg/g	40	135
13C-1,2,3,4,6,7,8-HpCDD	200	pg/g	40	135
13C-OCDD	400	pg/g	40	135

APPENDIX K-H

EXAMPLE FIELD FORMS

34

200

NO POUCH NEEDED.

See back for peel and stick application instructions.

FedEx Express USA Airbill

FedEx Tracking Number 8384 6513 9327

SPH41

Form I.D. No. 0215

Sender's Copy

1 From (Please print and press hard)

Date

Sender's FedEx Account Number 1482-5701-9

Sender's Name

Phone ( )

Company CONESTOGA-ROVERS & ASSOCIATES

Address 14496 N SHELDON RD STE 200

Dept./Floor/Suite/Room

City PLYMOUTH State MI ZIP 48170

2 Your Internal Billing Reference

First 24 characters will appear on invoice.

3 To

Recipient's Name

Phone ( )

Company

Address

To "HOLD" at FedEx location, print FedEx address. We cannot deliver to P.O. boxes or P.O. ZIP codes.

Address

Dept./Floor/Suite/Room

City State ZIP

Try online shipping at fedex.com

By using this Airbill you agree to the service conditions on the back of this Airbill and in our current Service Guide, including terms that limit our liability.

Questions? Visit our Web site at fedex.com or call 1.800.Go.FedEx® 800.463.3339.

0235804859

4a Express Package Service — Packages up to 150 lbs.

Delivery commitment may be later in more areas

- FedEx Priority Overnight — Next business morning
- FedEx Standard Overnight — Next business afternoon
- FedEx First Overnight — Earliest next business morning delivery to select locations
- FedEx 2Day — Second business day
- FedEx Express Saver — Third business day

FedEx Envelopes not available. Minimum charge: One-pound rate

4b Express Freight Service — Packages over 150 lbs.

Delivery commitment may be later in more areas

- FedEx 1Day Freight* — Next business day
- FedEx 2Day Freight — Second business day
- FedEx 3Day Freight — Third business day

*Call for Confirmation:

5 Packaging — *Declared value limit $500

- FedEx Envelope*
- FedEx Pak* — Includes FedEx Small Pak, FedEx Large Pak, and FedEx Sturdy Pak
- Other

6 Special Handling — Include FedEx address in Section 3.

- SATURDAY Delivery — Available ONLY for FedEx Priority Overnight and FedEx 2Day to select ZIP codes
- HOLD Weekday at FedEx Location — NOT Available for FedEx First Overnight
- HOLD Saturday at FedEx Location — Available ONLY for FedEx Priority Overnight and FedEx 2Day to some locations

Does this shipment contain dangerous goods? One box must be checked.

- No
- Yes — As per attached Shipper's Declaration
- Yes — Shipper's Declaration not required
- Dry Ice — Dry Ice, 9, UN 1845 ____ x ____ kg

Dangerous Goods (including Dry Ice) cannot be shipped in FedEx packaging.

- Cargo Aircraft Only

7 Payment Bill to: — Enter FedEx Acct. No. or Credit Card No. below.

- Sender — Acct. No. in Section 1 will be billed
- Recipient
- Third Party
- Credit Card
- Cash/Check

FedEx Acct. No. / Credit Card No. Exp. Date

Total Packages Total Weight Total Declared Value† $ .00

†Our liability is limited to $100 unless you declare a higher value. See back for details.

FedEx Use Only

8 Release Signature — Signing authorizes delivery without obtaining signature.

By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.

447

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FIELD FORM FIGURE 1

TYPICAL AIRBILL
REMEDIAL INVESTIGATION/ FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
Moraine, Ohio

CRA

CONESTOGA-ROVERS & ASSOCIATES

JOB NAME: SOUTH DAYTON LANDFILL JOB NO: 38443

SAMPLE ID: _____

SAMPLE DATE: _____ TIME: _____

SAMPLER NAME: _____

MATRIX: _____ ANALYSIS: _____

_____

PRESERVATION: NONE ☐ HNO_3 ☐ HCl ☐ H_2SO_4 ☐ NaOH ☐

NaOH/ZnOAc ☐ MeOH ☐ Other: Iced to 4 ± 2° C

SAMPLE LABEL

NOTES:

1) SAMPLE LABELS TO BE FIRMLY AFFIXED TO SAMPLE CONTAINERS.

2) ALL SAMPLE LABELS TO BE COMPLETED USING WATER INSOLUBLE INK.

CRA

CONESTOGA-ROVERS & ASSOCIATES

14496 N. SHELDON ROAD, SUITE 200

PLYMOUTH, MI 48170

CUSTODY TAPE

FIELD FORM FIGURE 2

EXAMPLES OF SAMPLE LABEL AND CUSTODY TAPE
REMEDIAL INVESTIGATION/ FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
Moraine, Ohio

CRA
CONESTOGA–ROVERS & ASSOCIATES, INC.
14496 Sheldon Road, Suite 200
Plymouth, MI 48170 (734) 453–5123

CHAIN OF CUSTODY RECORD

SHIPPED TO (Laboratory Name):

REFERENCE NUMBER:

PROJECT NAME:

SAMPLER'S SIGNATURE: _____ PRINTED NAME: _____

SEQ. No.	DATE	TIME	SAMPLE No.	SAMPLE TYPE	No. OF CONTAINERS	PARAMETERS										REMARKS
TOTAL NUMBER OF CONTAINERS																

RELINQUISHED BY: 1. _____	DATE: TIME:	RECEIVED BY: 2. _____	DATE: TIME:
RELINQUISHED BY: 2. _____	DATE: TIME:	RECEIVED BY: 3. _____	DATE: TIME:
RELINQUISHED BY: 3. _____	DATE: TIME:	RECEIVED BY: 4. _____	DATE: TIME:

METHOD OF SHIPMENT: AIR BILL No.

White -Fully Executed Copy Pink -Shipper Copy
Yellow -Receiving Laboratory Copy Goldenrod -Sampler Copy

SAMPLE TEAM: _____

RECEIVED FOR LABORATORY BY: _____

DATE: _____ TIME: _____

FIELD FORM FIGURE 3

TYPICAL CHAIN-OF-CUSTODY RECORD
REMEDIAL INVESTIGATION/ FEASIBILITY STUDY
SOUTH DAYTON DUMP AND LANDFILL
Moraine, Ohio

38443-07(005)GN-DE003 SEP 29/2008

APPENDIX K-I

LAND SURVEY, BATHYMETRY SURVEY, AND GEOPHYSICAL INVESTIGATION LETTER WORKPLAN

CONESTOGA-ROVERS
& ASSOCIATES

651 Colby Drive, Waterloo, Ontario, Canada N2V 1C2
Telephone: (519) 884-0510 Facsimile: (519) 884-0525
www.CRAworld.com

May 9, 2008

Reference No. 038443

Karen Cibulskis
Remedial Project Manager
United States Environmental Protection Agency
Region V
77 West Jackson Boulevard
Mail Code SR-6J
Chicago, IL 60604

Dear Karen:

Re: Final Land Survey, Bathymetry Survey, and Geophysical Investigation
South Dayton Dump and Landfill Site, Moraine, Ohio (Site)

This Letter Work Plan presents the South Dayton Dump and Landfill Potentially Responsible Party Group's (PRP Group's) approach for a land survey, bathymetry survey, and geophysical investigation of the Site. The work will help address data gaps and provide information to aid in the completion of a Feasibility Study (FS). The work will also allow for identification of surveyed areas that may require additional investigation or consideration prior to the beginning of intrusive fieldwork.

The PRP Group has prepared this Letter Work Plan based on discussions between the PRP Group and USEPA in February 2008 and April 2008. The Letter Work Plan incorporates comments received from USEPA on April 8, 2008.

SURVEYING

The objectives of the Site Survey are as follows:

- conduct a complete topographical survey of the entire Site by aerial photometry;
- survey locations of existing structures and features such as access roads, buildings, building foundations, fences monitoring wells, etc.;
- establish benchmarks for future surveying uses including but not limited to Site settlement monitoring;
- generate a current Site plan for use in future investigation and remedial alternative evaluation activities; and

- generate an accurate topographical map of the Site for use in determining current Site drainage patterns and for use in evaluating various landfill cap designs.

All survey work completed throughout this project will be performed by a State of Ohio registered land surveyor.

Survey data will be collected to obtain current topographic information in the area of the Site as bounded by the Great Miami River (GMR) to the north and west, Dryden Road to the east, East River Road to the southeast and Parcel 3264 to the south. The topographical survey will be completed utilizing aerial survey techniques. The Site was flown over April 2, 2008. Ten targets were placed on the ground in the survey area to act as control points for ground truthing the survey. In additional the horizontal locations of all boreholes, test trenches, test pits, monitoring wells, gas probes and staff gauges will be surveyed by ground personnel and reported in Ohio State Plane Grid Coordinates and in Decimal Degrees and elevations will be verified against the closest USGS benchmark monuments. Elevations will be surveyed according to the 1988 North American Vertical Datum (NAVD 88) for vertical coordinates and the 1983 North American Datum (NAD 83) for horizontal coordinates. Horizontal locations will be surveyed to the nearest 0.5-foot accuracy. Elevations for all monitoring well reference points (new and existing) will be surveyed to the nearest 0.01-foot accuracy. Elevations for all other locations will be surveyed to the nearest 0.1-foot accuracy. Five settlement monuments will be established within the PRP Group's preliminary direct contact risk presumptive remedy area in the central portion of the landfill on Lot 5177 for future use in settlement monitoring. The settlement monuments will be surveyed to the nearest 0.01-foot accuracy. The settlement monument locations are provided on Figure 1. Additional settlement monuments may be required in other areas of the Site. The need for additional settlement monuments will be discussed with USEPA as further data with respect to the location of landfill materials are obtained.

SURFICIAL METALLIC DEBRIS COLLECTION/STAGING

Prior to completing a geophysical investigation at the Site, CRA will retain a contractor to collect surfical metallic debris, empty drums and/or drum carcasses previously observed along the central access road and other areas across the Site, as necessary. The contractor will relocate this material to a central staging area located on-Site for interim storage in order to minimize its impact on the geophysical investigation. This debris will be managed as part of future waste characterization and consolidation activities, which will be conducted prior to implementing a remedy at the Site. Drums that are intact and have liquid or solid contents and are visually

determined to be in poor condition will be left in place. The location and contents (based on visual observation) of these drums will documented in a logbook and also marked on a Site plan. The location of drums left in place will be surveyed with a global positioning system (GPS) receiver, and reported in Ohio State Plane Grid Coordinates and in Decimal Degrees. These drum locations will be referenced to the same coordinate system used for the geophysical investigation, to allow surface metal locations to be easily matched to the geophysical maps.

The staging area will first be surveyed using the geophysical techniques identified below before it is constructed or used. After the geophysical investigation of the staging area, a staging pad will be constructed. The staging area will be installed with a containment berm and a 20-mil synthetic liner for leak and spill protection. The staging area will be located on Lot 5177 within the fenced in area of the Site for security purposes. Once debris collection is completed the area will be covered with polyethylene sheeting to prevent the accumulation of storm water within the area.

More than one staging pad may be constructed depending on how much debris is relocated. A typical staging cell construction detail is presented on Figure 2.

BATHYMETRY SURVEY

The objectives of the bathymetry survey are as follows:

- generate topographical information for the bottom of the Quarry Pond; and
- generate information for use in future investigation and remedial alternative evaluation activities.

A bathymetry survey will be completed to define the bottom of the Quarry Pond utilizing an echosounder attached to a GPS receiver to maintain control of sub-meter positioning. The bathymetry data and survey line locations will be stored in a digital format using Bathylog (or equivalent) software. The Echosounder and GPS will be programmed to collect respective data at 0.5- to 1.0-second time intervals. The bathymetry survey will be completed along pre-determined survey lines spaced 40 feet apart and oriented in an approximate north-south direction, which have been uploaded into the associated navigational software. Bathymetry data will also be collected on cross-lines oriented in an approximate east-west direction, and spaced 150 feet apart. Survey data will be used to complete a map of the Quarry Pond. Based on the results of the bathymetry survey an electromagnetic (EM) or magnetometer survey of the

Quarry Pond will be completed to identify metallic anomalies (i.e., drums) on the bottom of the Quarry Pond.

Land versions of the EM and magnetometer will be used if the pond is shallow, and marine versions will be used if the pond is deep. Specifically, if the pond is less than 10 feet deep, EM61 and magnetometer surveys will be completed using raft-mounted land instruments. If the pond is between 10 and 20 feet deep, EM31 and magnetometer surveys will be completed using raft-mounted land instruments. If the pond is between 20 and 30 feet deep, the EM survey will be completed using a marine EM instrument or an EM instrument with a 30-foot-depth of investigation; the magnetometer survey will be completed using raft-mounted land instruments. If the pond is more than 30 feet deep, EM and magnetometer surveys will be completed using marine instruments.

GEOPHYSICAL INVESTIGATION

The objectives of the geophysical investigation are as follows:

- identify buried metals and objects at the Site at surveyed locations; and
- identify Site areas which may require additional investigation.

The investigation will use magnetic, EM, and ground penetrating radar (GPR) techniques to identify both ferrous and non-ferrous buried metal objects at surveyed locations to depths of up 20 feet below ground surface. The magnetic survey will consist of total field and vertical gradient data collection. Magnetic field readings will be recorded at a background base station location during the course of the survey, to allow for correction of diurnal variation (i.e., magnetic drift), if necessary. The EM surveys will utilize an EM31-MK2 instrument (or equivalent), operating simultaneously in metal detection and conductivity modes, and an EM61 buried metal detector.

The EM61 survey will be used to detect the presence of buried metal objects in the shallow subsurface. The EM61 is a time domain instrument, which has an effective depth of investigation of approximately 10 feet below ground surface (bgs), and operates at a frequency of 150 Hz. The EM61 exhibits good lateral (or horizontal) resolution of buried metal objects (in the presence of one object or several objects situated in close proximity) in comparison to other EM methods, due to its stacked coil configuration. The coil separation for the EM61 is one foot. The EM31 survey will used to detect the presence of buried metal objects in the deeper subsurface. The EM31 is a frequency domain instrument, which has an effective depth of

investigation of approximately 17 feet bgs when carried at hip level and operating in horizontal dipole mode. The EM31 exhibits good lateral (or horizontal) resolution for individual buried metal objects but in situations where two or more objects are in close proximity to each other, the EM31 cannot delineate individual responses. This limitation can be attributed to the location of transmitter and receiver coils at either end of a 13-foot long cylindrical boom. The EM31 operates at a frequency of 9.8 kiloHertz (kHz).

The depth of investigation of a GPR survey is inversely proportional to the frequency of the instrument. That is, the higher the frequency, the more rapidly the GPR signal will attenuate or dissipate in the subsurface. Thus, the GPR survey will utilize a Ramac Rough Terrain Concept (RTC) low frequency system, with a 100-Mhz antenna (or equivalent) to optimize the depth of investigation. This instrument is characterized by an in-line transmitter-receiver antenna configuration, which allows for relatively rapid GPR data acquisition in comparison to other instruments. The EM and GPR surveys will also identify buried conduits or pipelines at the Site, the locations of which will be recorded for future reference. Conduits can include electric, communication, water, and gas lines as well as sewers and field tiles. Smaller conduits, however, may not be seen in surveyed areas where the ground is mostly clay with a high moisture content. The usefulness of GPR at this Site may be limited by any heterogeneity of landfilled materials and uneven terrain. It may be difficult to determine whether GPR survey results have been affected by de-coupling (bouncing) on the ground or signal scatter due to the ground matrix.

The areas of the Site in which the geophysical investigation will occur are presented on Figure 3. It should be noted that existing material storage piles located on and adjacent to the Valley Asphalt and Custom Delivery properties (Lots 5054 and 5177) and existing building structures at the Site (Lots 4610, 5054, 5171, 5172, 5173, 5174, and 5175) will physically limit the extent of the geophysical survey to be conducted. Minor amounts of brush and tree cutting will be required to facilitate the geophysical survey. Survey lines will be cleared to a minimum width of 4 feet, to facilitate geophysical surveying activities and to provide good GPR contact, or coupling. The in-line transmitter-receiver configuration will ensure adequate coupling is maintained during the GPR survey, since the width of the antennas along the geophysical survey lines is relatively narrow (approximately 6 inches wide). Any cleared brush will be removed from the survey lines to mitigate slip, trip and fall hazards, and to facilitate progress of the geophysical surveys.

Prior to conducting the surveys, a grid consisting of parallel lines will be established over the area of investigation (shown on Figure 3). The grid will utilize a number of control points that will be surveyed for horizontal and vertical location at 150-foot intervals in the approximate north-south direction, and 160-foot intervals in the approximate east-west direction.

Geophysical survey lines spaced 40 feet apart will be established between the control points, and will be designated with a Cartesian coordinate system as required by instrument data loggers. In addition, perpendicular (approximate east-west) geophysical survey lines will be established at 150-foot intervals, along the lines joining the control points. Magnetic, EM, and GPR measurements will be recorded at 0.5 second time intervals or, at a minimum, 0.7-foot distance intervals along these grid lines, and stored automatically in data loggers.

The anticipated vertical beam widths or effective investigative depths for the EM31 and EM61 are approximately 17 feet bgs (at hip level) and 10 feet bgs, respectively, as specified by the manufacturer. The vertical beam width or effective depth of investigation for the GPR survey will be dependant on conditions encountered in the field and will be evident on the trace plots, once compiled. The horizontal beam width of these three surveys (EM31, EM61, and GPR) is relatively poor, and will generally be restricted to the trend of the geophysical survey line and immediate surrounding area (i.e., 2 to 3 feet off-line). The magnetometer survey is a passive geophysical method; therefore, beam width is perhaps not the most appropriate term in describing the radius of detection for this instrument. The concentration of magnetic flux of the induced field in a buried object is a function of the magnetic susceptibility of the buried object, the degree of degradation (i.e., rusting out) it has undergone, and the size and orientation of the buried object. However, magnetometer surveys can typically yield an anomaly on the order of several hundred nanoTeslas (nT) over objects such as drums buried approximately 20 feet bgs. In addition, the lateral or horizontal resolution of a magnetometer survey is good, whereby an elevated magnetic response can be observed 10 to 20 feet adjacent to the buried object.

The purpose of the proposed geophysical investigation will be to act as a "screening tool", by providing potential targets for intrusive work based on anomalous metal detection responses. As such, it is impossible to speculate what the nature or composition of any suspected metal detection targets are, or how many may be present, until the anomalies are excavated and ground-truthed. Further, the configuration of the instrument (EM31) or measured quantity (magnetic field) of some geophysical instruments precludes identification of individual buried metal objects when two or more of these objects are present adjacent to, or in close proximity to each other, both vertically and horizontally. The only exception whereby discrete anomalies in close proximity to each other may be delineated is with the EM61 survey results, in the shallow subsurface.

The data loggers will be referenced to the Site survey grid, and will not be tied into GPS automatically. This will allow for more rapid data acquisition and data assessment on-Site (since the coordinates will already be in a Cartesian system and won't require conversion from latitude/longitude). This will also allow for more accurate locating of anomalous responses along the geophysical survey lines. Following completion of the geophysical investigation, the

location and extent of anomalous responses will be surveyed with a GPS system, and reported in Ohio State Plane Grid Coordinates and in Decimal Degrees (i.e., the same approach for the drums left in place).

The magnetometer, EM31, EM61, and GPR land surveys will be carried by operators, without the aid of a mobile system such as an ATV.

The geophysical investigation results will be presented as colored, contoured plots. The results will be used to finalize the locations of test pits and trenches.

The surface geophysical investigation will consist of collecting data on 40-foot spaced grid lines with intermediate 20-foot spaced grid lines over anomalous areas. The decision to perform 20-foot grid spacings will be evaluated at a minimum on a weekly basis on-Site, following a preliminary data assessment, which is scheduled to occur on the weekends or on rain days. The 20-foot grid spacings will be surveyed immediately following this evaluation, or following brush-clearing of the 20-foot grid lines, where required. CRA will discuss the results with USEPA and Ohio EPA's Site representative(s) as the work progresses; however, to accommodate the schedule, CRA does not intend to discuss the preliminary results with USEPA and Ohio EPA before starting 20-foot grids/concluding the survey.

Contour plots will be provided at appropriate intervals and color scales, to clearly accentuate anomalous responses.

The geophysical instruments used to collect the geophysical data will include:

- GEM GSM-19 Overhauser Proton Precession Magnetometer (or equivalent such as EG&G Geometrics G-858G cesium vapor magnetometer) to collect total magnetic field and magnetic gradient data;
- Geonics, Inc. EM-31 Ground Conductivity Meter to collect quadrature (terrain conductivity) and in-phase (metal detection) data;
- Geonics, Inc. EM-61 Buried Metal Detector to collect focused metal detection data; and
- Ramac RTC low frequency GPR system with a 100-Mhz antenna.

Instrument descriptions and survey procedures are provided in Attachment A.

The various geophysical surveys will be completed concurrently, to the extent practicable. Only one instrument of each type will be operated at any given time, to avoid potential interference

of multiple signals, especially in the case of the EM31. Further, a minimum distance of 150 feet will be maintained between instruments at any given time, to avoid another source of potential interference.

An EM or magnetometer survey of the Quarry Pond will be conducted after the completion of the bathymetry survey. The bathymetry survey of the Quarry Pond will allow for the proper selection of the geophysical survey equipment, based on the depth of the water column. The Quarry Pond geophysical survey will be completed using a GPS to ensure complete and effective coverage of the area has been completed. The survey will be conducted using a small boat with an outboard motor towing a non-metallic raft with the GPS and survey equipment. Specifically, the non-metallic raft will be constructed of wood, or other non-conductive material such as fiberglass.

The data will be used to identify areas of the Site that may require further investigation as part of the soil or groundwater sampling programs (under separate cover).

All work will be performed in accordance with the Field Sampling Plan, and Site Specific Health and Safety Plan pending USEPA's approval of these documents. Prior to conducting the work, local utility location services will be contacted to locate any known utilities.

SCHEDULE

The land survey, bathymetry survey, and the geophysical investigation work will be initiated within fifteen days of USEPA approval of this Letter Work Plan. These field tasks will be conducted concurrently and will be completed within an eight-week period of time. The PRP Group will provide the USEPA with verbal notification of field activities at least 15 days in advance of the initiation of field activities. Data processing, plotting, and drafting requirements for the bathymetry survey, land geophysics surveys, and waterborne surveys will take six weeks to complete, after which draft plots of the survey results will be provided to the PRP Group.

REPORTING

An updated Site plan and topographical map, and AutoCAD files with coordinates will be provided to the USEPA within one month of completion of the proposed work (i.e., the aerial photometry survey and survey of existing features (e.g. monitoring wells, surface water gauges, etc.). A preliminary topographical map will be provided to USEPA one month after the

completion of the aerial photometry survey. The topographic map will be prepared with a 10-foot contour interval. The contour interval may be adjusted if Site conditions warrant the use of a finer interval. Geophysical and bathymetry reports will be forwarded to the USEPA within two weeks of the PRP Group's receipt of the reports.

A map showing known and found utilities and other conduits will be provided concurrent with the geophysical and bathymetry reports.

Should you have any questions on the above, please do not hesitate to contact us.

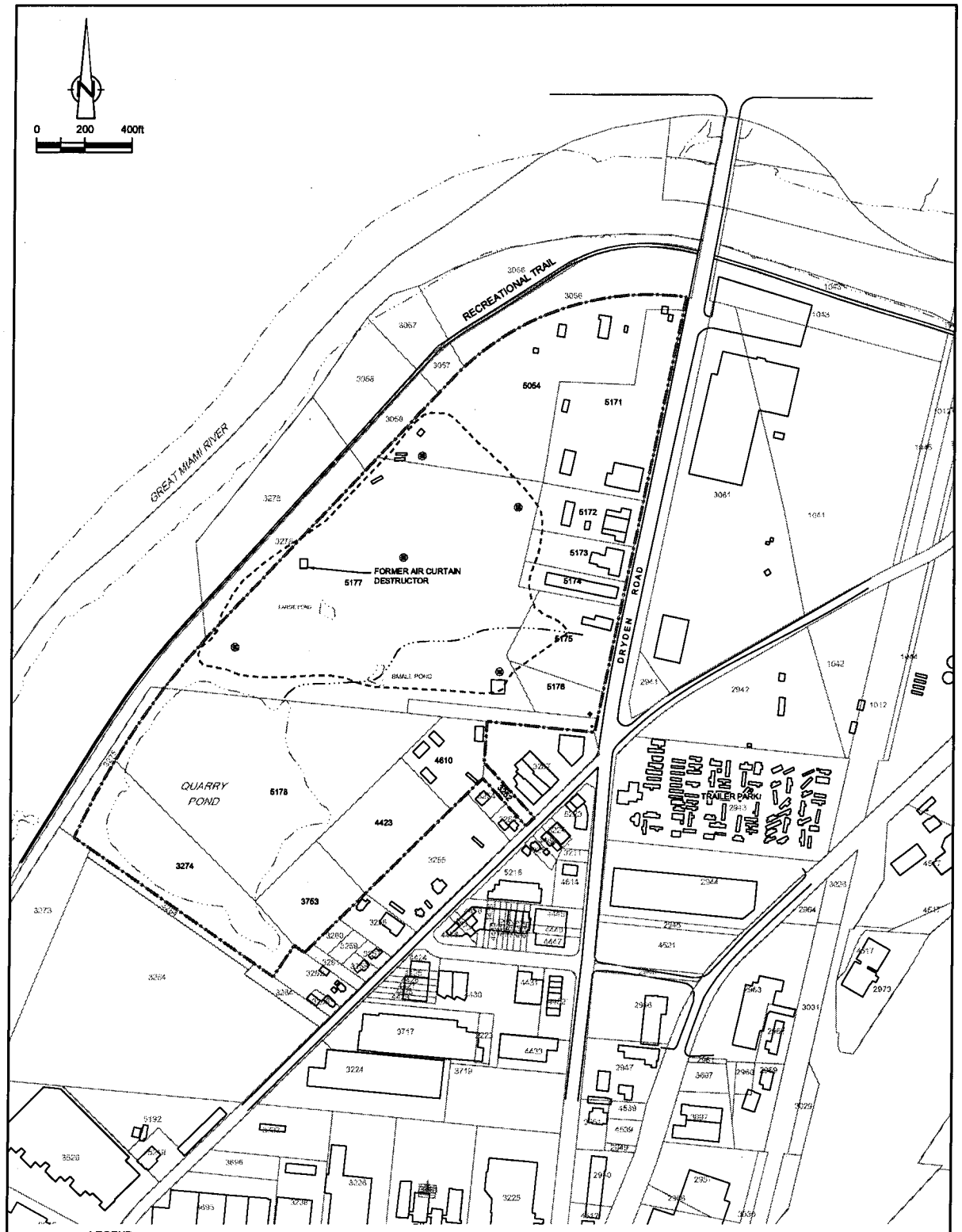
Yours truly,

CONESTOGA-ROVERS & ASSOCIATES

Stephen M. Quigley

LA/ca/28
Encl.

c.c. Matt Mankowski, USEPA (PDF)
Matt Justice, Ohio EPA (PDF)
Eric Kroger, CH2M Hill (PDF)
Scott Blackhurst, Kelsey Hayes Company (PDF)
Wray Blattner, Thompson Hine (PDF)
Ken Brown, ITW (PDF)
Jim Campbell, Engineering Management Inc. (PDF)
Tim Hoffman, Representing Kathryn Boesch and Margaret Grillot (PDF)
Paul Jack, Castle Bay (PDF)
Robin Lunn, Mayer Brown (PDF)
Roger McCready, NCR (PDF)
Karen Mignone, Pepe & Hazard (PDF)
Adam Loney, CRA (PDF)



LEGEND

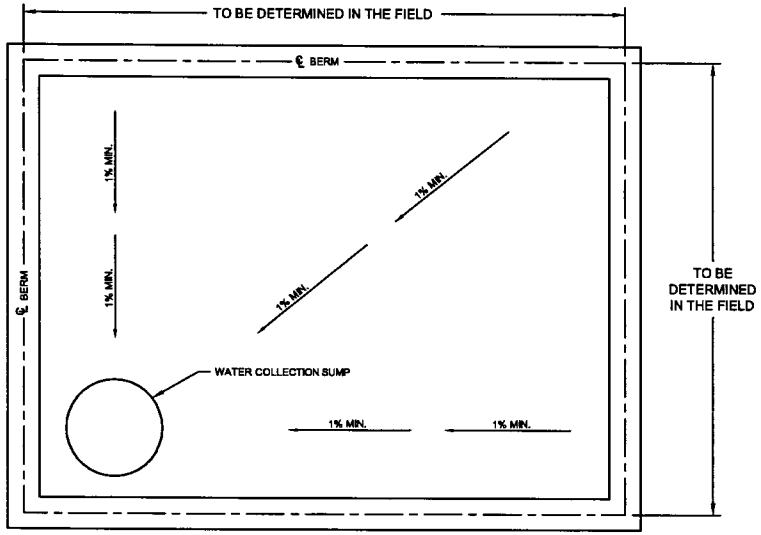
- SITE BOUNDARY (SOW 2006)
- PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA
- PARCEL BOUNDARY
- 3264 LOT NUMBER
- EDGE OF WATER
- EXISTING STRUCTURE
- PROPOSED SETTLEMENT MONUMENT LOCATION

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.

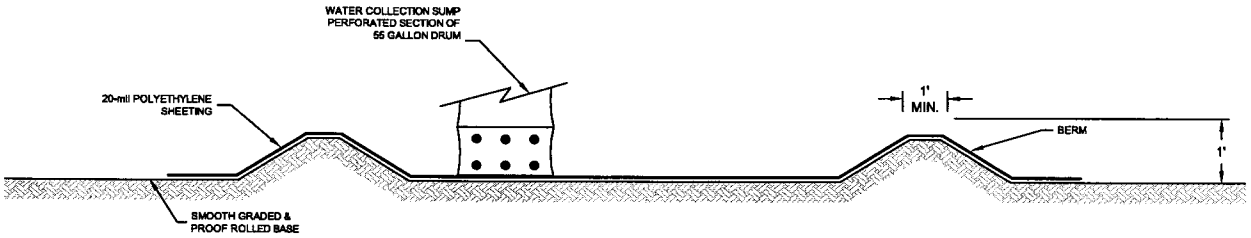
figure 1

PROPOSED SETTLEMENT MONUMENT LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-00(CIBU028)GN-WA001 APR 18/2008



PLAN VIEW

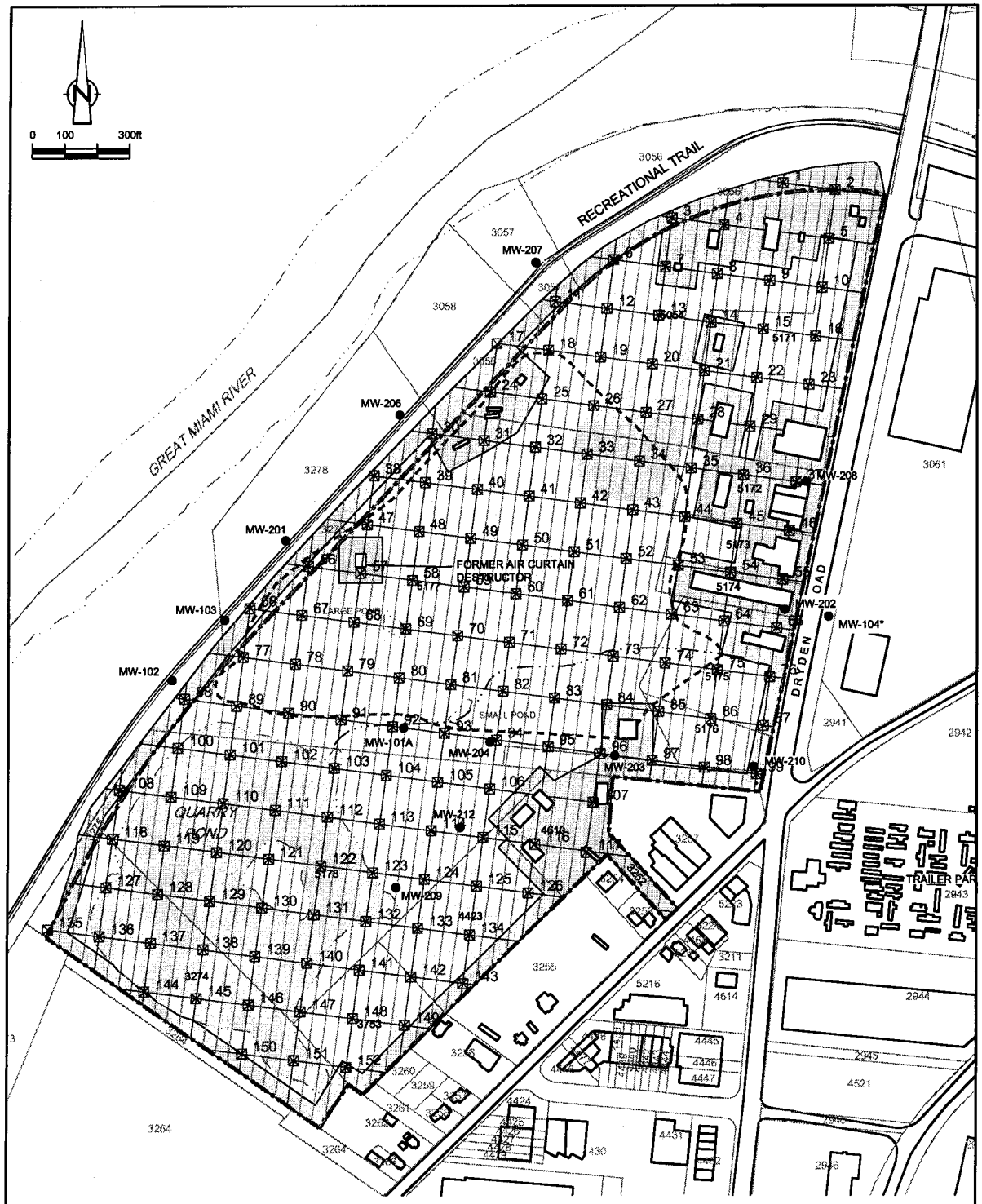


PROFILE

figure 2

STAGING PAD
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-00(CIBU028)GN-WA002 MAR 14/2008



LEGEND

MW-206 ● INTERMEDIATE ZONE MONITORING WELL LOCATION

SITE BOUNDARY (SOW 2006)

PRELIMINARY DIRECT CONTACT RISK PRESUMPTIVE REMEDY AREA

EDGE OF WATER

APPROXIMATE LOCATION

PROPOSED CONTROL POINTS AND GRID LINES

PROPOSED GEOPHYSICAL SURVEY COVERAGE

PROPOSED BATHYMETRIC AND WATERBORNE GEOPHYSICAL SURVEY COVERAGE

PROPOSED GEOPHYSICAL SURVEY COVERAGE WHERE FEATURES MAY MASK SURVEY RESPONSE

figure 3

PROPOSED CONTROL POINTS
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-00(CIBU028)GN-WA004 APR 18/2008

ATTACHMENT A

INSTRUMENT DESCRIPTION AND SURVEY PROCEDURES

ATTACHMENT A

INSTRUMENT DESCRIPTION AND SURVEY PROCEDURES

Instrumentation Description and Survey Procedures

The magnetometer records total magnetic field data from two sensors, top and bottom. The difference in total magnetic field between the two sensors divided by the vertical distance between the sensors equals the magnetic gradient. Magnetometers detect the presence of ferro-metallic objects, and are capable of lateral resolution of anomalies (i.e., anomalous responses are often observed adjacent to the buried object in addition to directly over the object). This allows for greater line and station spacings, and relatively rapid coverage of an investigative area. During the course of the survey, repeat readings will be recorded at a base station location situated away from any source(s) of magnetic interference to assess the degree of naturally-occurring diurnal variation (i.e., magnetic drift).

The EM31 consists of transmitter and receiver coils located at opposite ends of a 14-foot long boom. In vertical dipole mode, this coil configuration yields an approximate depth of penetration of 20 feet. As indicated by the manufacturer of the EM31, Geonics Limited (www.geonics.com), the effective depth of investigation for the EM31 is 6 metres, or approximately 20 feet below ground surface (bgs) in horizontal dipole mode. At hip level, this depth decreases to approximately 17 feet bgs. The EM31 is capable of operating simultaneously in both terrain conductivity and metal detection modes. The EM31 will be utilized in metal detection mode, since this instrument is capable of inducing secondary fields in all conductive buried metallic objects. Terrain conductivity readings will also be measured, in order to delineate areas of conductive fill.

EM31 metal detection anomalies can be characterized by two types of responses. Large anomalies covering a relatively wide area are identified by elevated responses, whereas smaller anomalies are characterized by very low (negative responses). The explanation of these results can be attributed to the 14-foot separation between the transmitter and receiver coils of the EM31. When sources of anomalies are much larger than the 14-foot coil spacing, the signal received by the EM31 becomes saturated, resulting in an elevated reading. When an object is smaller than the 14-foot coil spacing, the secondary field induced in the object opposes the primary field, yielding a negative resultant field (expressed as a percentage of the primary field). Both elevated and negative metal detection anomalies indicative of buried metal will be identified in the EM31 survey results.

The EM61 is a time-domain buried metal detector that consists of two rectangular transmitting and receiving coils in a stacked configuration, connected to a data logger. The coils measure approximately 1.5 by 3 feet, and are mounted to a wheeled cart. The transmitting coil emits 150 EM pulses per second into the ground at each measuring point. During the off time between transmitted pulses, receiver coils measure the decay of the transient electrical currents induced by the pulses. Electrical currents in moderately conductive earth materials (including moist clays, mineralized soils, etc.) dissipate rapidly, leaving only the more prolonged currents due to buried metal objects. The EM61 detects and measures the prolonged transient currents, yielding a result in millivolts (mV) proportional to the metallic content of the buried object, and inversely proportional to its depth of burial. Due to its stacked coil configuration, the EM61 is less susceptible to potential sources of interference including parked vehicles, fence lines, staged drums, power lines, etc. The EM61 survey will be completed along the survey lines by automatically triggering a reading at 0.7-foot stations. The effective depth of penetration of the EM61 is approximately 10 feet.

The Ramac™ Rough Terrain Concept (RTC) low frequency GPR system transmits at 100 Mhz, and is characterized by an in-line transmitter-receiver antenna configuration. GPR systems utilize pulsed EM waves, which are emitted from a transmitting antenna. They are propagated into the ground, and travel at velocities determined by the electrical properties of earth materials. As a GPR wave moving downward in the subsurface hits a buried object or boundary with different electrical properties, part of the wave energy is reflected back to the surface and is detected by a receiving antenna. The reflected wave is stored digitally, and processed as a trace of signal versus amplitude. As the antennas are moved along a survey line, a series of traces are recorded at discrete points. When presented collectively, these traces display a profile of the subsurface. The depth of subsurface penetration is directly dependent upon the frequency of the GPR system, and the conductivity of the soil. Signal attenuation is greater for higher frequencies, and also greater for conductive soils.

The geophysical survey procedures will be as follows:

1. The geophysical survey grid setup will commence with surveying of the control points at 150-foot intervals in the north-south direction and 160-foot intervals in the east-west direction, as indicated on Figure 3. Concurrently, brush clearing will commence between these control points, to facilitate additional grid setup described below;
2. The geophysical survey grid will be set up such that survey lines are spaced 40 feet apart. Wooden survey stakes labeled with the grid coordinates will be placed at 150-foot intervals along each gridline via surveying. Horizontal locations will be surveyed relative to the Ohio State Plane Grid Coordinates and Decimal Degrees. Elevations will be surveyed relative to NAVD 88. Horizontal locations will be surveyed to the nearest 0.5-foot accuracy relative to NAD 83. Elevations will be surveyed to the nearest 0.1-foot accuracy;
3. Geophysical data will be collected using a data logger on each geophysical instrument. The data recording for the magnetometer, EM31, EM61 and Ramac GPR system will be initiated for each station by the operator pressing the recording button. The station spacing for the magnetometer and EM31 will be approximately 5 feet, and will be

determined via pacing. The EM61, will be utilized in wheeled mode, and will automatically trigger the data logger to record a reading at 0.7-foot intervals.

4. The magnetometer survey will also include the use of a base station to determine diurnal variation. The base station(s) will be set up in area(s) free of ferromagnetic waste and base station readings will be recorded several times a day during the course of the survey. Base station readings will be recorded at a minimum of every 4 hours, to verify that the diurnal variation in the earth's magnetic field is negligible (i.e. <50 nT). Solar forecasts will be reviewed on a daily basis and in instances where increased solar activity is forecast, the magnetic survey will be temporarily suspended;

5. Data reduction will include downloading from the data loggers to a computer. The downloaded data will be processed for location. Magnetometer data may be corrected for diurnal variation, if required; and

6. The data will be contoured using SURFER® (Golden Software, Inc.). Separate contour plots for each data type will be prepared. Manual interpretation of the plots will be performed to assess the identified anomalies. This interpretation will include identification of anomalous areas for further investigation.

The 20-foot grid spacing will be completed for anomalies exhibiting the following responses: an in-phase response of 10ppt above background for the EM31, a metal detection response of 500 mV above background for the EM61, and a total magnetic field response of 500 nT above background for the Overhauser magnetometer. Geophysical and bathymetry reports will be forwarded to the USEPA within two weeks of the PRP Group's receipt of the reports. The decision to perform 20-foot grid spacings will be evaluated at a minimum on a weekly basis, following a preliminary data assessment which is scheduled to occur on the weekends or on rain days. Grid spacings at intervals less than 20 feet are presently not being considered. Thus, an anomalous response that is detected along a trend between 2 or more adjacent survey lines will be considered to be continuous between these adjacent survey lines.

The GPR data processing will consist of background (noise) removal, application of low- and high-pass filters, and Automatic Gain Control (AGC) gain to optimize the response of the GPR traces. GPR reflectors suspected of representing buried metal drums will be interpreted on the basis of a characteristic arch-shaped response.

APPENDIX K-J

ANALYTICAL DATA QUALITY ASSESSMENT AND VALIDATION SOP

PROPRIETARY DOCUMENT

ANALYTICAL DATA QUALITY ASSESSMENT AND VALIDATION – STANDARD OPERATING PROCEDURE (SOP)

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ACRONYMS

A	Acid Fraction of SVOC
B/N	Base/Neutral Fractions of SVOC
CCAL	Continuing Calibration Standard
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain of Custody Form
COV	Coefficient of Variance
CRA	Conestoga-Rovers & Associates
CRDL	Contract Required Detection Limit
CVAA	Cold Vapor Atomic Absorption
DL	Dilution
%D	Percent Difference
DQO	Data Quality Objectives
DUP	Duplicate
EDD	Electronic Data Deliverables
EQL	Estimated Quantitation Limit
FDV	Full Data Validation
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HT	Holding Time
ICAL	Initial Calibration Standard
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP/MS	Inductively Coupled Plasma/ Mass Spectrometry
ICS	Interference Check Sample
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IS	Internal Standard
LCS	Laboratory Control Spike
LCSD	Laboratory Control Spike Duplicate

ACRONYMS

MD	Matrix Duplicate
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
ND	Non-detect
NFG	National Functional Guidelines
PCB	Polychlorinated Biphenyls
QA	Quality Assurance
QC	Quality Control
QAPP	Quality Assurance Project Plan
RDV	Reduced Data Validation
REG 5	USEPA Region 5
RF	Response Factor
RRF	Relative Response Factor
RL	Report Limits
RPD	Relative Percent Difference
%RSD	Percent Relative Standard Deviation
SAP	Sampling and Analysis Plan
R^2	Correlation Coefficient
SOP	Standard Operating Procedures
SVOC	Semi-Volatiles Organic Compounds
TAL	Target Analyte List
TCL	Target Compound List
VOC	Volatile Organic Compounds
USEPA	United States Environmental Protection Agency

1.0 INTRODUCTION

The purpose of this Standard Operating Procedure (SOP) is to ensure that all analytical data quality assessment and validation (data validation) is performed by Conestoga-Rovers & Associates (CRA) in a consistent manner and in accordance with project-specific requirements. The purpose of data validation is to ensure that only those data that meet project-specific data quality objectives (DQO) are used for project decision making and reporting.

This SOP discusses data validation of the parameters associated with the following analytical techniques:

- Gas Chromatography/Mass Spectrometry (GC/MS) - volatile organic compounds (VOC) and semi-volatile organic compounds (SVOCs);
- GC - polychlorinated biphenyls (PCBs), chlorinated pesticides, VOCs, herbicides, etc;
- High Performance Liquid Chromatography (HPLC) - polynuclear aromatic hydrocarbons (PAH), carbamates, explosives, etc;
- Spectrometric - including inductively coupled plasma (ICP), inductively coupled plasma/mass spectrometer (ICP)/MS), cold vapor atomic absorption (CVAA);
- Spectrophotometry (Spec) - cyanide, sulfate, phenolics and other inorganics;
- Ion Chromatography (IC) - inorganic anions; and
- Titrimetric - chloride, etc.

The data validation procedures detailed in this SOP are based on guidance and quality control procedures established by the United States Environmental Protection Agency (USEPA) including the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", USEPA-540/R-99/008, October 1999 and the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", USEPA 540/R-94/013, February 1994, collectively referred to as NFGs throughout this SOP. Selected guidance for ICP/MS data review relies on the latest inorganic NFG, "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", USEPA 540/R-01/008, July 2002 referred to as NFG-2. In addition to the federal guidance data review follows other project specific guidance such as regional SOPs; analytical methods, and laboratory SOPs as defined in project

documentation (Quality Assurance Project Plan (QAPP), Sampling and Analysis Plan (SAP), Work Plan and Purchase Orders).

2.0 LEVELS OF ANALYTICAL DATA REVIEW AND ASSESSMENT

CRA completes several types or levels of data review and assessment including compliance assessment and data validation. Descriptions of these and the information reviewed are discussed below.

As defined by the USEPA, data verification is the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual compliance[1]. Data validation is an analyte-specific and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance to determine the analytical quality of a specific data set[1].

This SOP addresses three levels of data validation, application of each is dependent on project-defined data quality objectives. For all samples sets, regardless of validation level employed, a compliance assessment must be completed. The compliance assessment includes an assessment of data package completeness and compliance with project requirements as outlined in the project Analytical Scope of Work.

The three levels of validation are as follows, the specific elements addressed for each level of review are presented in Table 2.1.

Reduced data validation (RDV), also referred to as data verification or Level III validation, entails the review of sample preservation and holding time compliance, as well as the lab and field quality control results (blank, spikes, surrogates, etc). It does not include the assessment of raw data, instrument calibration data, internal standard recoveries, interelement interference check standards, nor serial dilutions.

Full data validation (FDV), also referred to as Level IV validation, includes all steps of the RDV process as well as the review of raw data associated with sample and quality control results and confirmation of analyte quantitation and identification (e.g., sample calculations, mass spectra, chromatograms, etc) on a minimum of 10 percent of the laboratory data. FDV also includes the review of raw data and results for instrument

1 United States Environmental Protection Agency (USEPA) Guidance on Environmental Data Verification and Data Validation, USEPA QA/GA-8, November 2002.

calibration, (instrument performance, initial and continuing calibration), internal standards, interelement interference check standards, and serial dilutions.

A third level of validation is identified in the Region III document "Innovative Approaches to Data Validation, 1995". This level of validation (referred to as innovative approach) falls somewhere between FDV and RDV; it does not include the review of raw data, but it does include the assessment of all sample and QC results, including instrument calibration data, internal standard recoveries, interelement interference check standards, and serial dilutions.

For clients requesting FDV on 10 percent of their data, and a lesser validation level on the remaining data packages, the innovative approach is often used on the remaining 90 percent to ensure consistent qualifier application (this approach is referred to as 90/10). If FDV and RDV are used in this manner, the problem is that the package assessed using FDV may have calibration outliers and therefore data qualifications, while the same outliers would not yield data qualification in a RDV (e.g., calibration results are not assessed in RDV).

The innovative approach is similar to that used for projects requiring validation of New Jersey reduced deliverables.

Be aware that project-specific requirements may include data validation levels that differ from the three main types outlined above.

3.0 ELECTRONIC DATA FORMAT AND CODING

Data are received from the laboratory in hardcopy format and electronically as an EquIS® 4-file electronic data deliverable (EDD). A 10 percent manual check is performed to ensure that the EDD matches the hardcopy data. The data are imported into the database by the database analysts. For projects requiring data validation, the chemist is provided with an electronic output form the database (a flatfile workbook) for their use in reviewing the data and for communicating data qualifications back to the database analysts for import into the database.

The flatfile workbook consists of a sample summary worksheet, a flatfile, and a xtab.

3.1 SAMPLE SUMMARY WORKSHEET

The sample summary provides a snapshot of the type and number of sample results imported into the database for that sample set. This spreadsheet indicates the number of analytes reported for each sample/test as shown in the example below:

Sample_Name	*MADEP-VPH*	*SW-6010R*	*SW-7470R*	*SW-8015V*	*SW-8021 BTEX*	*SW-8260 BMTBE*
027626-032707-MW1					4	
027626-032707-MW11					4	
027626-032707-MW11 DUP					4	
027626-032707-MW12					4	
027626-032707-MW2					4	
027626-032707-MW4					4	
027626-032707-MW7					4	
027626-032707-MW8					4	
27626-051707-COMP1		7	1			
27626-051707-SB10 (10-12)				1		5
27626-051707-SB10 (14-15)				1		5
27626-051707-SB10(8-10)				1		5
27626-051707-SB2 (10-12)	3			1		5
27626-051707-SB4 (14-15)				1		5
27626-051707-SB4 (4-6)				1		5
27626-051707-SB4 (8-10)				1		5
27626-051707-SB5 (10-12)				1		5

The analytical Scope of Work and Chain of Custody or Field Key can be used to check that the proper number of analytes were reported and that the samples were analyzed for the required tests and that the proper number of results were reported.

If the sample summary shows that there were 26 VOCs reported for all samples except for one sample that had 24, the discrepancy should be checked.

This spreadsheet will summarize the number of results flagged as "reportable" by the laboratory. If the laboratory performs a dilution or reanalysis, they are directed to include both results in the EDD, but one should be flagged as "Y" for reportable and the other "N". If the lab has failed to flag data for dilutions or re-analyses properly, the sample summary may show double the number of analytes for one or more of the samples. This is rectified by changing the reportable flags in the flatfile from "Y" to "N" for the appropriate results.

3.2 FLATFILE WORKSHEET

Any changes to be made to the database by the chemist are inputted into the flat file and imported back into the database.

3.2.1 FLATFILE FIELDS

The following are a list of fields included in the flatfile with descriptions.

sys_sample_code	sample code based on the Sample ID, including suffix for dilution and reanalysis
test_surrogate_key	sample/method code
cas_rn	analyte code based on CAS registry number including suffix for total or dissolved metals
lab_anl_method_name	analytical method code including suffix for listed parameters (i.e., Toxicity Characteristic Leaching Procedure [TCLP]) or holding time assuming one method produces multiple holding times
lab_prep_method_name	sample preparatory method code

lab_sample_id	laboratory sample ID
chemical_name	compound/analyte
loc_name	sample location
sample_name	sample identification number
parent_sample_code	"sys_sample_code" of parent sample for field duplicate (FD)/matrix spike (MS)/matrix spike duplicate (MSD)/laboratory replicate (LR)/laboratory control sample duplicate (LCSD)
sample_matrix_code	sample matrix code
sample_type_code	original (N), field duplicate (FD), Trip Blank (TB),etc
start_depth	initial sample depth
end_depth	final sample depth
depth_unit	sample depth unit
sample_delivery_group	Laboratory SDG
instrument_id	instrument identifier
column_number	both columns are required when testing for PCBs and pesticides
test_batch_id	unique identifier for all lab batches
analysis_date	analysis date
analysis_time	analysis time
sample_date	sample collection date
dilution_factor	sample dilution factor
prep_date	sample prep date
result_value**	positive detection value (null if non-detect)
method_detection_limit**	MDL (unadjusted)
reporting_detection_limit**	report limit
	this field name is misleading, the values in this field are normally the sample-specific Practical Quantitation Limits (PQL) for organics and wet chemistry, and either the Contract Required Detection Limit (CRDL) or instrument detection limit (IDLs) for metals, depending on lab convention.
quantitation_limit**	sample-specific MDL/IDL - this field name is misleading, it has nothing to do with the quantitation limit.
result_unit**	units
lab_qualifiers**	laboratory qualifiers
validator_qualifiers**	validation qualifiers
detect_flag	**"Y" for positive detection, "N" for non-detects

qc_rpd_status	used to indicate whether the relative percent difference was within control limits
approval_a**	validation reason code
approval_b	DV guidance document code
collection_quarter	collection quarter
approval_code**	validation level code
organic_yn	indicates whether test was organic ("Y") or inorganic ("N")
result_type_code	Result type - "TRG" for a target or regular result, "TIC" for tentatively identified compounds, "SUR" for surrogates, "SC" for spiked compounds, or "IS" for internal standards (internal standards are not required in the deliverable)
sampling_reason	sample event identifier
result_comment	import metadata
fraction_code	test category of the analysis method. (e.g., VOCs, SVOCs, Wet, etc.)
percent_moisture	percent moisture of the sample portion used in this test; this value may vary from test to test for any sample
subsample_amount	amount of sample used for the test
subsample_amount_unit	unit of measurement for "subsample_amount"
final_volume	final sample volume
final_volume_unit	unit of measurement for "final_volume"
qc_original_conc	The concentration of the analyte in the original (unspiked) sample
qc_spike_added	The concentration of the analyte added to the original sample
qc_spike_measured	The measured concentration of the analyte
qc_spike_recovery	The percent recovery for the spike
qc_dup_original_conc	The concentration of the analyte in the original (unspiked) sample
qc_dup_spike_added	The concentration of the analyte added to the original sample
qc_dup_spike_measured	the measured concentration of the analyte in the duplicate
qc_dup_spike recovery	The duplicate percent recovery
qc_rpd	The relative percent difference between the spike and the spike duplicate
qc_spike_lcl	Lower control limit for spike recovery
qc_spike_ucl	Upper control limit for spike recovery
qc_rpd_cl	Relative percent difference control limit

qc_spike_status	Flag used to indicate whether the spike recovery was within control limits
qc_dup_spike status	Flag used to indicate whether the duplicate spike recovery was within control limit
reportable_result	Indicates whether sample result is to be reported - "Y" will allow for extraction, "N" will suppress

Fields that are color-coded are included for the chemist's information only. These fields should not be modified, as any changes will not be imported back into the database. If any changes are needed for these fields, please notify the database analyst.

Do not copy the data to another spreadsheet as data values *will* change at the discretion of the software. If copying data is necessary:

i) open a new spreadsheet;

ii) select entire spreadsheet;

iii) "Format/Cells/Number(tab)/Text";

iv) return to source spreadsheet and "Copy"; and

v) return to destination spreadsheet and "Paste Special/Text".

The user must select the entire spreadsheet prior to sorting a column, otherwise the data in column will sort within itself.

The flatfile provided should be in the "Autofilter" mode which enables the user to filter the data to view only those desired fields (e.g., view VOCs only by selecting 8260 in the method field, or view a single SDG, etc.).

The flatfile can be made more manageable by "hiding" columns not needed. To do this, right click on the column letter to highlight the entire column select "hide". You can highlight one or several columns before hiding them. To unhide, highlight the two visible columns adjacent to the hidden columns, right click and select "unhide". To unhide all columns at once, click on the top left-corner of the .xls sheet, which highlights the entire sheet, and go to "format", "columns" and select "unhide".

3.2.2 CHECK FLAT FILE PROGRAM

The Check Flatfile tab is located at the top of the flatfile worksheet includes three macros to be used on the flatfile including Convert Time, Run Flatfile Completeness Checker (covered later in this section), and Run Qualifier Copy (J, B, U).

Convert Time - All fields are submitted as text and must remain as text (to preserve significant digits in the database) with the exception of the "analysis_time" field which can be converted to standard time format (for sorting purposes). Use the "convert time" command under the "Check Flat File" tab at the top of the spreadsheet.

Run Qualifier Copy - For data that are validated, the validator qualifier field must include all lab qualifiers that should be applied to the final data set. This includes any lab qualifiers that would be preserved through validation (e.g. "U"). Prior to adding validator qualifiers to the flatfile, this program is used to copy pertinent lab qualifiers to the validator qualifier field as follows:

- U Qualifier - The program will ask "copy over U qualifiers?". If the user selects "yes", the program finds all instances of U in the "lab_qualifiers" field and inputs a "U" into the "validator_qualifier" field. The macro also inputs "PLU" (Preserve Lab U) into the "approval_code" field.

  If the user selects "no", the program does not do anything with lab "U" qualifiers.
- J Qualifier - When the program finds a "J" in the "lab_qualifiers" field it copies "J" into the "validator_qualifiers" field, and copies "BRL" (Below Reporting Limit) into the "approval_code" field. Note that some laboratories use the "J" flag to denote inorganic method blank contamination. Be sure to review inorganic "J" flags to ensure the flag reflects a value less than the reporting limit, and not method blank contamination. This is particularly true for TestAmerica Inc. The definition of the inorganic "J" flags must be verified by reviewing the laboratory report.
- B Qualifier – Most labs use the B qualifier differently for organic and inorganic tests, for organics, a B typically denotes blank contamination and for inorganics, a B typically denotes that a positive detection is less than the reporting limit (similar to how a "J" qualifier is used for organics). When the program finds a "B" in the "lab_qualifiers" field, it then looks at the "organic_yn" field. If the value in that field

is an "N", then it copies "J" into the "validator_qualifiers" field, and copies "BRL" into the "approval_code" field, if the value of the "organic_yn" field was a "Y", then it does nothing; no B is copied over.

3.2.3 DATA QUALIFICATION

Data qualifiers are inputted into the "validator_qualifier" field. Each "validator_qualifier" must have an associated reason for the data qualification that is inputted into the "approval_a" code field (see Section 5.0 for more information on reason codes). The addition of new valid values for reason codes must be submitted to Julie Lidstone for review and inclusion in the "approval_a" reference table.

All preserved laboratory qualifiers in the "validator_qualifier" field must also have an associated "approval_a" code. The "approval_a" code for the preserved lab qualifier of organic "J" and inorganic "B" is "BRL". The "approval_a" code for preserved laboratory qualifier "U" is "PLU".

To qualify for blank contamination, the value in the "result_value" field is removed, the "detect_flag" field is changed from "Y" to "N", the U qualifier is inputted into the "validator_qualifiers" field, the "quantitation_limit" field (remember, this field contains the sample-specific detection limit) is raised to the detected value, and the appropriate reason code is added to the "approval_a" field (e.g., MBK). If the original sample concentration was an estimated value (e.g., 6J), the "reporting_limit" field for the non-detect value will remain unchanged. If the analyte's concentration was not an estimated value, the value in the "reporting_limit" field must be elevated to the original sample concentration.

Samples may have more than one set of results due to dilutions and re-analyses completed by the laboratory. The EDD includes a field titled "reportable_result" which is populated with either "N" or "Y" for each result. When multiple analyses are performed, the laboratory will designate which result they feel is most usable based on quality control data. Any time there are multiple analyses for a sample, the data validator must determine which analysis best meets the project DQO. Consideration should be given to all quality control (QC) factors (e.g., surrogate recoveries, holding

times, internal standard recoveries, etc.), during this determination. If the validator chooses to report a result other than that selected by the lab, the "reportable_result" field is changed from "Y" to "N" for the value that the validator wishes not to report, and the "reportable_result" field is changed from "N" to "Y" for the value to be reported.

Due to the fact that some validation guidance documents include different qualifiers and vary in the application of qualifiers, each result in the database must include documentation of the guidance used for data validation. This information is stored in the "approval_b" field (see Section 4.0 for more information on "approval_b" codes); this field must be populated for all results.

All data in the database must show the level of validation applied. The "approval_code" value represents the level of validation. The "approval_code" will be defaulted to "4" upon opening the spreadsheet and must be revised after validation to the "approval_code" field prior to submittal for import into the database. Approval codes are summarized in Table 3.1.

Non-detect reporting limits are normally reported from the "reporting_detection_limit" field of the flatfile, which is typically the PQL for organics and the IDL or CRDL (depending on lab reporting convention) for inorganics.

If it is required that reporting limits other than those stored in the "reporting_detection_limit" field are required (e.g., IDLs be used for metals in place of CRDLs, or IDLs and MDLs across the board for risk assessment, or TRRP reporting), the reporting limits can be stored in an optional field called "custom_field_2". Prior to flatfile generation, the validator will inform the database analyst about where reporting limits for non-detect data are to be obtained and these values will be imported into the "custom"field"2" field of the flatfile.

If custom_field_2 is used, when changing reporting limits due to method blank concentrations, the validator must be sure to change the values in the "custom_field_2", "reporting_detection_limit" and "quantitation_limit" fields as appropriate.

3.2.4 FLATFILE COMPLETENESS CHECKER

This program is located under the Check Flatfile tab and is used to check the final flatfile for consistency and completeness. For example, the checker will identify inconsistencies like results that have "U" listed under "validator_qualifier" field, and a "Y" populated in the "detect_flag" field. This checker must be run and all errors addressed before submitting the flatfile back to database for import into the database.

3.2.5 SAVING THE UPDATED FLATFILE

Upon completion of validation, the "L" in the file name is changed to a "V" and the validator's initials are added to the end of the file name. The "peer" review person may also add their initials to the end of the file name.

3.3 XTAB GENERATION

Under the check flatfile tab, there is an option to prepare a crosstab (xtab) table of results. This table can be used by the chemist to review the final data and ensure that there are no missing results and that data appear to be reported correctly.

4.0 REVIEW OF APPLICABLE QUALITY CONTROL DOCUMENTS AND STANDARDS

Prior to commencing data validation, all applicable QC documents must be reviewed and understood. Where applicable, this includes the QAPP, DQO, region-specific or project-specific validation guidelines, permits, SOPs, and project SAPs, etc.

Unless otherwise specified, data validation is performed using the principles outlined in the USEPA NFG and the QC criteria outlined in the analytical methods. Project-specific quality documents will take precedence over all other guidance documents for decision-making purposes during analytical data review and assessment.

The data validation guidance document used to perform the validation must be recorded in the database. A two-digit code has been assigned to each document (see Table 4.1) and the appropriate code must be entered into the "approval_b" field of the flatfile.

5.0 REASON CODE ASSIGNMENT

As data are qualified throughout the data validation processes, a reason code is assigned to document the reason for the qualification. A list of reason codes and definitions are presented in Table 5.1. These reason codes are stored in the database in the "approval_a" field. For all data qualifications, the appropriate reason code must be entered into the "approval_a" field of the flatfile.

When a data value is qualified for multiple reasons, up to three reason codes (9 characters) can be assigned appending each to the primary qualifying code without separation. For example, if a data point is qualified for outlying surrogate and laboratory control sample (LCS) recoveries, the result is qualified with "LCSSUR".

6.0 DATA VALIDATION PROCEDURES

Data validation is performed in accordance with the USEPA NFGs (or other region- or project-specific validation guidance document) and any other project-specific quality control documents. The following sections detail the data validation procedures for each element.

During data validation, data qualifiers are applied to sample results to indicate potential biases in the data. The USEPA NFG data validation qualifier definitions are presented in Table 6.1. The CRA abbreviated data qualifier definitions are presented in Table 6.2. Either set of definitions can be used as appropriate based on agency and project-specific requirements.

6.1 DATA PACKAGE COMPLETENESS AND COMPLIANCE REVIEW

Prior to data quality assessment and validation, the data package must be checked to ensure that all elements are present. Data package completeness and compliance checklists are included as Appendices A-1 and A-2.

6.2 SAMPLE PRESERVATION AND HOLDING TIME ASSESSMENT

Holding times are defined as the amount of time that elapses between the collection of the appropriately preserved sample from the source in the field and the beginning of the analysis procedure (which may include extraction and a separate holding time period from extraction to analysis). The published sample container, preservation, and holding time requirements are presented in Table 6.3. Note that this table is to be used as guidance and could vary by method, region, agency, etc. If project-specific requirements differ from this table, project-specific requirements take precedence.

Sample holding times are verified by the Chain of Custody forms (COCs), laboratory custody records, and narrative to verify that samples were received at 4°C (±2°C) (if applicable), were properly preserved and analyzed within the method or project-specific holding times. For exceedances, qualify data as indicated in the following Sample Preservation and Holding Time (HT) Non-Compliance Action Table.

SAMPLE PRESERVATION AND HOLDING TIME NON-COMPLIANCE ACTION

		Action		
Assessment Element	*Failure*	*Non-Detects*	*Positive Results*	*Approval Code*
Holding Time [1]	Sample HT exceeds HT criteria by ≤2x (NFG) Sample HT exceeds HT criteria by >2x (NFG)	UJ R	J J	HTQ
Chemical Preservation	Not preserved in the field (NFG) (pH adjustments by lab are acceptable)	UJ	J	SPV
Inappropriate Container	Sample collected in inappropriate container (CRA)	Prof. Judgment	Prof. Judgment	SPV
Headspace (VOC Waters)	VOC vial contained headspace (CRA) (> pea-sized air bubble)	UJ	J	SPV
Sample/Cooler Temperature [2]	No Ice (CRA) and >6 and ≤10°C (CRA) No Ice (CRA) and >10 and ≤20°C (CRA) No Ice (CRA) and >20°C (CRA)	Prof. Judgement UJ R	Prof. Judgement J J	SPV SPV SPV

Notes:
1 Holding time criteria will incorporate professional judgement for certain parameter/matrices.
2 Sample/cooler temperature evaluation will include other considerations, such as the presence or absence of ice in the cooler, the number of days in transit and the specific parameters requested. Same day delivery of samples is exempt from receipt temperature requirements.

6.3 INSTRUMENT PERFORMANCE CHECK - ORGANICS BY GC/MS AND METALS BY ICP/MS

Tuning is a quality control performance check that is particular to the use of instruments having a Mass Spectrometer (MS) as the detector. The MS functions by bombarding the target analytes with electrons as they enter the analyzer. The electrons collide with target analyte molecules causing them to ionize. The analyzer then performs a count of the ion abundance of each ion created with the compound molecules. The software used in conjunction with the mass analyzer prepares a plot of abundance versus mass of the ions, called a mass spectrum. The relative abundance of the ions created and detected from the compound molecules is dependent upon the electrical and magnetic properties of the mass analyzer.

The following items are evaluated as part of the instrument performance check during FDV with calculations spot-checked at a 10 percent frequency:

- Check tune summaries to ensure that instrument tune checks were performed every 12 hours (organics); and that all investigative samples are accounted for within those tune windows.
- Verify the tune checks were analyzed five times consecutively (ICP/MS only). If tune checks have not been performed at the required frequency, use professional judgement to determine whether associated sample results are valid.
- Check that all abundances are normalized to the proper m/z.
- Verify the ICP/MS percent relative standard deviation (%RSD) values were within the specified criteria.
- Check that the proper number of significant figures have been reported.
- Spot-check for transcription errors between raw data and tune summary form.
- Recalculate some of the relative ion abundance's to verify lab's calculations.
- Verify that spectra were obtained using accepted background subtraction techniques.
- Verify that appropriate (method- or QAPP-specific) ion abundance criteria were used.

If errors are noted, contact the laboratory and request a corrected re-submittal. If the lab cannot resubmit data, all associated data should be rejected (R).

Check all ion abundances against appropriate criteria. If outliers are noted, use professional judgement to determine whether the data are usable. If data are judged to be usable, the data validation report must clearly state the nature of the tune non-conformances. If the tune non-conformances are judged to impact data quality, qualify data as follows:

INSTRUMENT TUNE CRITERIA AND VALIDATION ACTION

		Action		
Assessment Element	*Criteria*	*Non-Detects*	*Positive Results*	*Approval Code*
VOC BFB Criteria (NFG)	50 8-40% of 95 75 30-60% of 95 95 Base Peak (100%) * 96 5-9% of 95 * 174 50-120% of 95 * 175 4-9% of 174 * 176 93-101% of 174 * 177 5-9% of 176 * 173 <2% of 174	* m/e non-compliant UJ (NFG)	* m/e non-compliant J (NFG)	TUN
	Incorrect base mass assignment	R (CRA)	R (CRA)	TUN
SVOC DFTPP Criteria (NFG)	51 30-60% of 198 *68 <2% of 69 69 mass of 69 *70 <2% of 69 127 40-60% of 198 *197 <1% of 198 *198 Base Peak (100%) *199 5-9% of 198 275 10-30% of 198 365 >1% of 198 *441 Present but <443 *442 >40% of 198 *443 17-23% of 442	* m/e non-compliant UJ (NFG)	* m/e non-compliant J (NFG)	TUN
	Incorrect base mass assignment	R (CRA)	R (CRA)	TUN
Metals (NFG)	Analyzed 5 times consecutively	R (NFG)	R (NFG)	TUN
	Peak Width to 0.75 amu @ 5% peak height	UJ (NFG)	J (NFG)	TUN
	m/e ±0.1 amu over range of 6- 210 amu	UJ (NFG)	J (NFG)	TUN
	%RSD absolute signals <5%	UJ (NFG)	J (NFG)	TUN
Timeframe (NFG)	> 12 hour timeframe but ≤13 hours (from injection of tuning standard) (CRA)	UJ (CRA)	J (CRA)	TUN
	>13 hour timeframe	R(CRA)	R(CRA)	TUN

6.4 INSTRUMENT CALIBRATION - ORGANICS

The following sections address initial and continuing instrument calibration for organic analyses. Instrument calibration data are reviewed during FDV only.

6.4.1 INITIAL INSTRUMENT CALIBRATION - ORGANICS

Calibration is the establishment of a quantitative relationship between the response of the analytical procedure and the concentration of the target analyte. The initial calibration (ICAL) is the procedure that functions as the calibration curve for the target analytes. A necessary prerequisite is that a confident identification of the target analyte has already been established.

To assess ICAL data:

- Check lab data to ensure that initial calibration standards were analyzed at the proper frequency.
- Verify a minimum of five standards were utilized and were analyzed at the appropriate concentrations.
- Check that the ICAL was analyzed prior to all samples associated with it.
- Verify that the ICAL met the minimum average relative response factors (RRF) and %RSD criteria identified in the NFG unless alternate method criteria are specified.
- Check lab data to ensure that the compounds identified were within the retention time (RT) criteria.
- Verify that at least three peaks were used in the calibration of each Aroclor during PCB analysis.

If the calibration standards were not analyzed at the proper frequency, use professional judgement to determine the usability of the data.

- Spot check calculations (10 percent) for RRF or response factors (RF) for methods not using internal standards.
- Spot-check calculations (10 percent) of the calibration curve. If various calculation methods were used (e.g., average RRF, linear regression, quadratic, etc.), results reported using each calculation must be verified.
- For GC methods, spot-check the calculation of retention time windows.
- Spot-check %RSD, correlation coefficient (R^2) calculations if used.

If errors are identified, contact the lab to have the data package corrected and resubmitted.

- Check the linearity or the calibration curve (%RSDs $\leq$30 percent for GC/MS, %RSDs $\leq$20 percent for GC, R^2 values >0.99).
- For methods using internal standards, check analyte sensitivity; average RRF must be $\geq$0.05.

If linearity or sensitivity criteria are not met, qualify data as follows:

ORGANIC INITIAL CALIBRATION CRITERIA AND VALIDATION ACTION

Method	*Assessment Element*	*Criteria*	*Failure*	*Action*		*Reason Code*
				Non-Detects	*Positive Results*	
GC/MS (VOC & SVOC)	Minimum Average Relative Response Factor (RRF) (NFG)	0.05 (NFG)	<0.05	R	J	ICL
	Relative Standard Deviation (%RSD) (NFG)	≤30.0%	>30.0%	UJ (CRA)	J (CRA)	ICL
	Quadratic Coefficient of Determination (R^2) (SW846)	≥0.99 (SW846)	<0.99 (SW846)	UJ (CRA)	J (CRA)	ICL
	Linear Correlation Coefficient (R) (SW846)	≥0.995 (CRA)	<0.995	UJ(CRA)	J(CRA)	ICL
GC & HPLC (PCB, Pest., VOCs, SVOCs) [1]	% Relative Standard Deviation (%RSD) (SW-846) or	≤ 20% (SW-846)	>20%	UJ [2]	J [2]	ICL
	Linear Correlation Coefficient (R) (SW846)	≥0.995 (CRA)	<0.995	UJ [2] (CRA)	J [2] (CRA)	ICL

Notes:
1 Multi-component analyte %RSD limits are based on average of all peaks used in calibration.
2 CRA modification of NFG qualification:
- Aroclor 1016 Non-compliance is representative of Aroclor 1221 through 1248
- Aroclor 1260 Non-compliance is representative of Aroclor 1254 through 1268

6.4.2 CONTINUING INSTRUMENT CALIBRATION - ORGANICS

The continuing calibration verification (CCAL) is used to verify that the initial calibration is maintained and correct while the instrument is used to process samples. The CCAL also serves to determine that the identification criteria are still being met. Valid sample results will always be preceded by an acceptable CCAL analysis for GC/MS methods and are bracketed by acceptable CCAL analyses for GC methods.

- Check lab data to ensure that continuing calibration standards were analyzed at the proper frequency.
- Verify that calibration standards were at the appropriate concentrations.
- Compare %Drift values to those compounds quantitated utilizing a quadratic curve.

- Check that the GC/MS CCAL met the minimum RRF and maximum percent difference (%D)/%Drift criteria.

If calibration standards were not analyzed at the proper frequency, use professional judgement to determine the usability of the data.

- Spot-check calculations (10 percent) for RRF or RF for methods not using internal standards.
- Spot check calculations (10 percent) for RRF or RF, and %D/%Drift.
- For GC, verify that the analyte retention times fall within the established retention time windows.

If errors are identified, contact the lab to have the data package corrected and resubmitted.

- Check the linearity (%D/% Drift $\leq$25 percent (GC/MS), %D $\leq$15 percent GC)
- For methods using internal standards, check analyte sensitivity; average RRF must be $\geq$0.05

If linearity or sensitivity criteria are not met, qualify data as follows:

ORGANIC CONTINUING CALIBRATION CRITERIA AND VALIDATION ACTION

Method	*Assessment Element*	*Criteria*	*Failure*	*Action*		*Reason Code*
				Non-Detects	*Positive Results*	
GC/MS (VOC & SVOC)	Minimum Average Relative Response Factor (RRF) (NFG)	≥0.05 (NFG)	<0.05	R (NFG)	J (NFG)	CCL
	Percent Difference (%D) (NFG)	≤25% (NFG)	> 25%	UJ (NFG)	J (NFG)	CCL
	Percent Drift (%Drift) (SW846)	≤25% (SW846)	> 25%	UJ (CRA)	J (CRA)	CCL
GC & HPLC (PCB, pest, VOCs, SVOCs) 1	Percent Difference (%D) (NFG) or	≤15% (SW-846)	> 15%	UJ 2 (CRA)	J 2 (CRA)	CCL
	Percent Drift (%Drift) (SW846)	≤15% (SW846)	> 15%	UJ 2 (CRA)	J 2 (CRA)	CCL
	Retention Time Window SW846)	±0.07 Min of Standard (SW846)	Outside Window	Prof. Judgment (CRA)	Prof. Judgment (CRA)	CCL

Notes:
(1) Multi-component analyte %D and % Drift limits are based on average of all peaks or concentrations used in calibration.
(2) CRA modification of NFG qualification:
- Aroclor 1016 Non-compliance is representative of Aroclor 1221 through 1248
- Aroclor 1260 Non-compliance is representative of Aroclor 1254 through 1268

6.5 INSTRUMENT CALIBRATION - INORGANICS

The following sections address initial and continuing instrument calibration for inorganic analyses. Instrument calibration data are reviewed during FDV only.

6.5.1 INITIAL INSTRUMENT CALIBRATION - INORGANICS

Calibration is the establishment of a quantitative relationship between the response of the analytical procedure and the concentration of the target analyte. The initial calibration (ICAL) is the procedure that functions as the calibration curve for the target analytes. A necessary prerequisite is that a confident identification of the target analyte has already been established.

If required by the method or QAPP, the CRDL must be verified with a standard (CRI) at 2 times (x) the CRDL or 2x the instrument detection limit (IDL), whichever is greater.

- Check that the ICAL was analyzed prior to all samples associated with it.
- Check lab data to ensure that the ICALs were analyzed at the proper concentration and frequency.
- Verify the initial calibration verification (ICV) was analyzed at the proper concentration and frequency.
- Check the linearity coefficient of variance (COV) $\geq$0.995.
- Check lab data to ensure the CRI was analyzed at the proper frequency, if required.

If calibration standards were not analyzed at the proper frequency, use professional judgement to determine the usability of the data.

- Spot-check calculations for calibration curves at a frequency of 10 percent. Results using calibration curve must be verified.

If errors are identified, contact the lab to have the data package corrected and resubmitted.

INORGANIC INITIAL CALIBRATION CRITERIA AND VALIDATION ACTION

Method	*Assessment Element*	*Criteria*	*Failure*	*Action* [1]		*Reason Code*
				Non-Detects	*Positive Results*	
ICP, ICP/MS, CVAA, Spec., IC	COV (Metals, Mercury, Gen. Chem.)	≥0.995 (NFG)	COV < 0.995	Prof. Judgement (NFG)	J (NFG)	ICL
ICP, ICP/MS (Metals)	Initial Calibration Verification (ICV)	± 10% of true value (NFG)	< 90% but ≥ 75% < 75% > 110% but ≤125% > 125%	UJ (NFG) R (NFG) None None	J (NFG) R (NFG) J (NFG) R (NFG)	ICL ICL ICL ICL
CVAA (Mercury)	ICV	± 20% of true value (NFG)	< 80% but ≥ 65% < 65% > 120% but ≤135% > 135%	UJ (NFG) R (NFG) None None	J (NFG) R (NFG) J (NFG) R (NFG)	ICL ICL ICL ICL
Spec. & IC (Gen. Chem.)	ICV	± 15% of true value (NFG)	< 85% but ≥ 70% < 70% > 115% but ≤130% > 130%	UJ (NFG) R (NFG) None None	J (NFG) R (NFG) J (NFG) R (NFG)	ICL ICL ICL ICL
ICP, CVAA ICP/MS Spec. (Metals, Mercury Gen. Chem.)	CRI [2]	Beginning of Analysis (if applicable) ± 20% of true value (±30% for ICP/MS) (NFG-2)	< LCL but ≥ 50% < 50% > UCL	UJ (NFG-2) R (NFG-2) None	J (if <2x RL) (NFG-2) J (if <2x RL) (NFG-2) J (if <2x RL) (NFG-2)	CRA CRA CRA

Notes:
1 All samples prepared within the analytical batch are affected.
2 The 2002 NFG for inorganics will be used to evaluate the CRI standard if CRI data is required.

6.5.2 CONTINUING INSTRUMENT CALIBRATION - INORGANICS

The continuing calibration verification (CCV) is used to verify that the ICAL is maintained and correct while the instrument is used to process samples. The CCV also serves to determine that the identification criteria are still being met. Valid sample results will always be bracketed in time by acceptable CCV analyses.

- Check lab data to ensure that continuing calibration standards were analyzed at the proper frequency.
- Verify that calibration standards were at the appropriate concentrations.

If calibration standards were not analyzed at the proper frequency, use professional judgement to determine the usability of the data.

INORGANIC CONTINUING CALIBRATION CRITERIA AND VALIDATION ACTION

Method	*Assessment Element*	*Criteria*	*Failure*	*Action* [1]		*Reason Code*
				Non-Detects	*Positive Results*	
ICP, ICP/MS (Metals)	Continuing Calibration Verification (CCV)	After every 10 samples ±10% of true value (NFG)	<90% but ≥75% < 75% >110% but ≤125% >125%	UJ (NFG) R (NFG) None None	J (NFG) R (NFG) J (NFG) R NFG)	CCL CCL CCL CCL
CVAA (Mercury)	CCV	After every 10 samples ±20% of true value (NFG)	<80% but ≥65% < 65% >120% but ≤135% > 135%	UJ (NFG) R (NFG) None None	J (NFG) R (NFG) J (NFG) R (NFG)	CCL CCL CCL CCL
Spec. & IC (Gen. Chem.)	CCV	After every 10 samples ±15% of true value (NFG)	<85% but ≥70% < 70% >115% but ≤130% >130%	UJ (NFG) R (NFG) None None	J (NFG) R (NFG) J (NFG) R (NFG)	CCL CCL CCL CCL
Spec. (Gen. Chem.)	Distillation Check	Midrange CN standard distilled (±15% of undistilled std) (NFG) if applicable	>115% <85%	None UJ (NFG)	J (NFG) J (NFG)	CCL CCL

Notes:
1 All samples between non-compliant CCVs within the analytical sequence are affected.

6.6 BLANKS (METHOD BLANKS, INSTRUMENT BLANKS, CALIBRATION BLANKS AND FIELD BLANKS)

Blank samples are prepared, collected and analyzed to determine if contamination of samples is potentially introduced during the collection and analysis process. Sources of sample contamination can include the containers and equipment used to collect the samples, preservatives added to the samples, other samples in transport coolers and laboratory sample storage refrigerators, standards and solutions used to calibrate instruments, glassware and reagents used to process samples and the analytical instrument sample introduction equipment. Each area of analysis has its own particular suite of common laboratory contaminants.

Method blanks apply to all samples belonging to the same sample preparation batch, or in the case of VOC, all samples analyzed on the same instrument on the same day. Inorganic initial calibration blanks (ICBs) apply to all samples after while continuing calibration blanks (CCBs) apply to all samples analyzed immediately before and after. Field blanks (trip blanks, rinse blanks, equipment blanks, filter blanks, etc.) are intended to identify contamination introduced during sample collection and/or storage and should be applied only to those samples collected at the same time as, and with the same equipment as the field blanks (see sample collection note, COC, sample key or field personnel to determine which field blanks apply to which samples). Trip blanks are applied to all VOC samples transported to the laboratory in the same shipping cooler as the trip blank.

If blanks were not analyzed at the proper frequency, potential contamination cannot be assessed for the corresponding investigative samples. This should be noted in the data validation report.

- Check the extraction logs, run-logs, and method blank summary forms to ensure that method blanks were prepared and analyzed with samples at the required frequency (typically one method blank per analytical batch).
- Check the sample run-log to ensure that instrument blanks were analyzed after any sample having significantly high target analyte concentrations.
- Check the run-log to ensure that ICBs and CCBs were analyzed at the proper frequency for inorganic analyses (typically every 10 samples).
- Review blank raw data for target analytes.

If target analytes are detected in the blanks, detection of these analytes in associated investigative samples may reflect contamination, and must be qualified as indicated in the table below. For assessing contamination in soil samples, individual sample weights (dry or wet) must be applied to the blank result before comparing it to the sample result. Soil samples with associated field blank contamination less than the reporting limits will be documented in the validation text without qualification. Significant field blank contamination for soils will require qualification. Sample preparation and dilution

factors for laboratory blanks (ICBs, CCBs, and method blanks) must also be applied to the blank results.

For sample results qualified non-detect (U) to reflect contamination, change the "detect" field in the flatfile from "Y" to "N", raise the report limit (RL) to the sample analyte concentration (when above the RL) and delete.

BLANK VALIDATION ACTION

Assessment Element	*Failure*	*Action* [1]			*Reason Code*
		Non-Detects	*Positive Results (RL Reporting)*	*Positive Results (MDL Reporting)*	
Method Blank Contamination	Target analytes detected <5x blank value or <10x for common lab contaminants [2] (NFG)	NA	< RL qualify U at RL > RL qualify U at value	< RL qualify UJ at value > RL qualify U at value	MBK
ICB and CCB Contamination	Target analytes detected <5x blank (NFG)	NA	< RL qualify U at RL > RL qualify U at value	< RL qualify UJ at value > RL qualify U at value	CBK CBK
Field Blank Contamination	Target analytes detected <5x blank value or <10x for common lab contaminants (CRA) (waters only)	NA	< RL qualify U at RL > RL qualify U at value	< RL qualify UJ at value > RL qualify U at value	TBK, FBK, RBK, FIL

Notes:
1 Hierarchy of qualifying data due to blank contamination is MBK, CBK, TBK, FBK, RBK, FIL. (CRA)
2 Common laboratory contaminants include: VOC - acetone, 2-butanone, methylene chloride and cyclohexane and SVOC - phthalates.

6.7 SYSTEM MONITORING COMPOUNDS (SURROGATES) - ORGANICS

Surrogates are non-target compounds added to every sample at the beginning of the sample preparation to monitor the success of the sample preparation on an individual sample basis.

For PCB analysis tetrachloro-m-xylene (TCMX) the more volatile of the two surrogates, is more volatile than any of the target analytes of this method. The recoveries of TCMX are linked to the lowest volume to which the sample extract was concentrated and target compounds such as Aroclor 1221 and Aroclor 1232. Samples with higher weight PCBs

contain substantial portions of decachlorobiphenyl (DCB), which will effect the calculated recovery of this surrogate when these analytes are present in the sample.

- Check surrogate summary forms to verify that all investigative samples have been accounted for.
- Spot-check raw data to verify the recoveries on the surrogate summary form.
- Spot-check calculations of the surrogate recoveries.
- Check that the control limits used are the appropriate limits for the project.
- Check surrogate recoveries against the laboratory control limits.

If any errors are found in the surrogate summaries, or are determined in the calculations, contact the laboratory and request the forms and any affected data pages are resubmitted with corrections.

If outliers are noted, verify that samples were re-analyzed. If surrogate recoveries for extractable parameters are still outside of the control limits, verify that the laboratory re-extracted the samples to confirm matrix interference. When there are unacceptable surrogate recoveries, followed by acceptable re-analyses, verify that the designated successful analysis was reported in the flatfile.

Sample data with outlying surrogate recovery should be qualified as summarized below. SVOC surrogate recoveries are assessed by fraction (base/neutral or acid) and only those analytes within that fraction affected by outlying surrogate recoveries are qualified.

Interferences and/or sample dilutions can prohibit surrogate recovery assessment. In such cases, it should be noted in the data validation report that surrogate recoveries could not be assessed.

SURROGATE CRITERIA AND VALIDATION ACTION

Assessment Element	*Failure*	*Action*		*Reason Code*
		Non-Detects	*Positive Results*	
GC, GC/MS & HPLC (excluding SVOC analysis)	If one or More Surrogates:			
	%R > UCL (NFG)	None	J (NFG)	SUR
	%R <LCL but ≥10% (NFG)	UJ (NFG)	J (NFG)	SUR
	%R <10% (NFG)	R (NFG)	J (NFG)	SUR
	Diluted (≥ 1:5) (CRA)	None	None	None
GC/MS SVOC Surrogate Recovery	1 out in a fraction > 10%	None	None	SUR
	≥2 out of a fraction %R > UCL (NFG)	None	J (NFG)	
	≥2 out of a fraction %R <LCL but ≥10% (NFG)	UJ (NFG)	J (NFG)	SUR
	One %R <10% NFG)	R (NFG)	J (NFG)	SUR
	Diluted (≥ 1:5) (CRA)	None	None	None

If the surrogates in method blanks and/or laboratory control samples (LCS) are as low as sample surrogate recoveries, matrix effects cannot be assumed and the overall extraction efficiency is in question. The problem should be noted in the data validation notes, and the laboratory should be contacted to provide an explanation and corrective actions for future analyses.

6.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATES - ORGANICS

An MS consists of a sample fortified with a known amount of a target analyte and is typically analyzed in duplicate (MS/MSD). Analysis of the MS/MSD and comparison with the unspiked sample result provides the ability to assess accuracy and precision of the method on a given sample matrix. MS/MSD recoveries are used for a qualitative indication of accuracy due to matrix effects. The relative percent difference (RPD) between the recoveries is used for a qualitative indication of precision.

- Verify that MS/MSDs were prepared and analyzed at the proper frequency (typically one per sample batch, up to 20 samples).

If MS/MSDs were not prepared and analyzed at the proper frequency, note this in the data validation report.

- Spot-check MS/MSD recovery calculations from raw data.

- Spot check RPD calculations.

If errors are determined in the calculations, contact the laboratory and request that the forms and any affected data pages be resubmitted with corrections.

- Assess recoveries against control limits.

If outlying analyte recoveries are identified, qualify all associated results for that sample only as summarized below. MS/MSD outliers observed in the organic analyses are associated with the parent sample only. Outlying results for multiple MS/MSD sample recoveries can indicate a systematic error (e.g., recoveries for multiple MS/MSDs show similar biases). In such cases, results for all associated samples in that batch may be qualified.

In cases where the original sample concentrations are significantly greater than the spiking concentrations (>4x), matrix spike recoveries cannot be assessed. This should be noted in the data validation report, and the associated samples are not qualified.

- Assess RPD values against laboratory established control limits.

If outlying MS/MSD percent recoveries or RPDs are identified, qualify all associated positive results for that sample only as follows:

MS/MSD - ORGANICS VALIDATION ACTION

Assessment Element	*Failure* [1]	*Action* [2]		*Reason Code*
		Non-Detects	*Positive Results*	
MS/MSD Accuracy (Recovery)	%R > UCL (Reg. 5) (Both MS & MSD)	None	J	MSD
	%R <LCL but ≥10% (Reg. 5) (Both MS & MSD)	UJ	J	MSD
	%R <10% (Reg. 5) (Either MS or MSD)	R	J	MSD
MS/MSD Precision	RPD > Criteria (Reg. 5)	None	J	MSD

Notes:
1 List of target compounds the laboratory will utilize are presented with each sample delivery group. The criteria used in validation will be those limits statistically generated by the laboratory, and may change periodically. Therefore the limits used for validation are referred to as the upper control limit (UCL) or lower control limit (LCL).
2 CRA Modification to NFG Qualifications:
- Aroclor 1016 Non-compliance is representative of Aroclor 1221 through 1248
- Aroclor 1260 Non-compliance is representative of Aroclor 1254 through 1268

6.9 MATRIX SPIKE/MATRIX SPIKE DUPLICATES OR MATRIX SPIKE/MATRIX DUPLICATE - INORGANICS

An MS is a sample fortified with a known amount of a target analyte and is typically analyzed in duplicate (MS/MSD). Analysis of the MS/MSD and comparison with the unspiked sample results provides the ability to assess accuracy and precision of the method on a given sample matrix. Precision may also be assessed through analysis of a matrix duplicate (MD). MS/MSD recoveries are used for a qualitative indication of accuracy (bias) due to matrix effects. The RPD between the recoveries (MS/MSD) or the results (MD) is used to assess precision. Only project specific sample matrix spikes are considered when evaluating and qualifying samples.

- Verify that MS/MSD or MS/MD were prepared and analyzed at the proper frequency (typically one per sample batch).

If MS/MSD or MS/MD were not prepared and analyzed at the proper frequency, note this in the data validation notes.

- Spot-check MS/MSD recovery calculations from raw data (FDV only).
- Spot-check RPD calculations.

If errors are determined in the calculations, contact the laboratory and request that the forms and any affected data pages be revised and resubmitted.

- Assess recoveries against control limits.

If outlying analyte recoveries are observed, qualify results for all associated samples (samples prepared in the same analytical batch).

In cases where the original sample concentrations are significantly greater than the spiking concentrations (>4x), MS recoveries cannot be assessed. Since the accuracy cannot be adequately determined, accuracy is considered not calculable (NC).

- Assess RPD values against control limits. If control limits are not specified for the project, use 20 percent for water samples and 35 percent for soil samples with results greater than five (5) times the RL. For sample results less than five (5) times the RL, precision is assessed by comparing the difference between the two results to a control limit of plus or minus (±) the RL amount for water samples and ±2 times the RL amount for soil samples.
- If one result from the sample/MD set is non-detect, precision is calculated using the analyte reporting limit for the non-detect results.

If MS/MSD or MS/MD data do not meet the above criteria, qualify all associated positive results as follows.

MS/MSD & MD - INORGANICS VALIDATION ACTION

Assessment Element	*Failure* [1]	*Action*		*Reason Code* [2]
		Non-Detects	*Positive Results*	
MS or MS/MSD Accuracy (Recovery)	%R ≥125% (NFG) (MS and MSD)	NA	J	MSQ or MSD
	%R ≥30% and <75% (NFG) MS and MSD	UJ	J	MSQ or MSD
	%R <30% (NFG) MS or MSD	R	J	MSQ or MSD
MS/MSD or MD Precision	RPD (if both values are >5X RL, Waters >20%, Soils >35%	NA	J	MSD or DUP
	RL (if either value is <5X RL), Waters >1X RL value (Reg. 5), Soils >2X RL value (Reg. 5)	J	J	MSD or DUP

Notes:
1 Laboratory may develop specific ICP/MS criteria.
2 Use MSD reason code for any data qualifications related to MS/MSD accuracy and/or precision. Use the MSQ reason code for any data qualifications related to accuracy when MS only is analyzed. Use the DUP reason code for any data qualifications related to precision when MD is analyzed.

6.10 LABORATORY CONTROL SPIKE/LABORATORY CONTROL SPIKE DUPLICATE SAMPLES

A LCS consists of a portion of analyte-free water or solid phase sample that is spiked with target analytes at a known concentration. The LCS is processed through the entire method procedure with each sample batch; the results are examined for target analyte recovery. In analytical batches where an MS/MSD sample is not available the LCS may be analyzed in duplicate. The LCS/laboratory control sample duplicate (LCSD) can be used by the laboratory in cases where the MS/MSD failed to achieve acceptable recovery and/or precision. If the LCS/LCSD fails to generate acceptable results, this should cause concern about the validity of the results for all samples in the batch.

- Verify that LCS or LCS/LCSD were prepared and analyzed at the proper frequency (typically one per sample batch).

If LCS or LCS/LCSD were not prepared and analyzed at the proper frequency, note this in the data validation narrative.

- Spot-check LCS or LCS/LCSD recovery calculations from raw data (FDV only).
- Check that the control limits used are the appropriate limits for the project.

If errors are determined in the control limits or calculations, contact the laboratory and request that the forms and any affected data pages be resubmitted with corrections.

- Assess recoveries against control limits.

If outlying analyte recoveries are identified, qualify all associated sample results in that sample batch as summarized below. If the LCS or LCSD recoveries indicate a systematic error (e.g., recoveries for all spiking compounds are low), qualifications of all compounds (spiking and non-spiking compounds) can be performed.

LCS/LCSD VALIDATION ACTION

Assessment Element	*Failure* [1]	*Action* [2]		*Reason Code* [3]
		Non-Detects	*Positive Results*	
LCS or LCSD Accuracy	%R >UCL (NFG)	None	J	LCQ or LCD
	Organics %R ≥10% but <LCL (Reg. 5)	UJ	J	LCQ or LCD
	Inorganics %R ≥50% but < 79% (NFG)	UJ	J	LCQ or LCD
	Organics %R <10% (Reg. 5)	R	J	LCQ or LCD
	Inorganics %R < 50 % (NFG)	R	J	LCQ or LCD
LCS/LCSD Precision	RPD > Lab control Limit (CRA)	NA	J	LCD

Notes:

1 Organic LCS Criteria - List of target compounds the laboratory will utilize are presented with each sample delivery group. The criteria used in validation will be those limits statistically generated by the laboratory, and may change periodically. Therefore the limits used for validation are referred to as the upper control limit (UCL) or lower control limit (LCL).

2 CRA Modification to NFG Qualifications:
- Aroclor 1016 Non-compliance is representative of Aroclor 1221 through 1248
- Aroclor 1260 Non-compliance is representative of Aroclor 1254 through 1268

3 Use LCD reason code for any data qualifications related to LCS/LCSD accuracy and/or precision. Use LCQ reason code for any data qualifications related to accuracy when LCS only is analyzed.

6.11 INTERNAL STANDARDS

Internal standards (IS), assessed during FDV only, are used as the quantitation and relative retention time standard for the target analytes analyzed by MS. Low area counts for the internal standards translate into falsely high reported values for target

analytes in the samples. Anyreported value for an analyte is an estimate when high or low IS area counts are reported.

- Verify that all samples are accounted for on the IS review forms.
- Spot check for transcription errors between the IS area counts reported on the summary form, and the raw data from calibration and sample analyses.
- Spot check the IS area range calculations.
- Spot check IS recoveries for the samples.

If there are errors in the IS summaries, or errors are determined in the calculations, contact the laboratory and request that the forms and any affected data pages be resubmitted with corrections.

- Check IS recoveries against control limits of 50-200 percent (GC/MS) or 30-120 percent (ICP/MS). If outliers are noted, verify that samples were re-injected. Where there are unacceptable IS recoveries, followed by acceptable re-analyses, verify that the designated successful analysis was reported in the flatfile.
- Verify that all internal standards were recovered.
- Check that the IS brackets the masses of target analytes (ICP/MS).
- Verify when outliers are observed during ICP/MS analysis, a two fold dilution is performed and a reanalysis of the calibration blank is performed.
- For IS recoveries outside of the control limits, only results for those analytes calculated from the outlying ISs will be affected as follows:

INTERNAL STANDARD CRITERIA AND VALIDATION ACTION

Assessment Element	*Failure*	*Action*		*Reason Code*
		Non-Detects	*Positive Results*	
IS Recovery (GC/MS)	>200% (NFG)	NA	J	IST
	<50% but ≥25% (NFG)	UJ	J	IST
	<25% (NFG)	R	J	IST
IS Recovery (ICP/MS)	Sample IS >120% (NFG-2)	NA	J	IST
	Sample IS <30% (NFG-2)	UJ	J	IST
	CCB, CCV IS >120% (SW-846)	NA	J	IST
	CCB, CCV <80% (SW-846)	UJ	J	IST
	Reanalysis of CCB, CCV not performed for outliers (NFG-2)	R	R	IST

6.12 INTERFERENCE CHECK SAMPLE - METALS

The interference check samples ICS (ICSA and ICSAB), assessed during FDV only, are analyzed at the completion of daily calibration and at the end of operation (minimum of two analyses per 8 hours of run time). The ICS data should be carefully checked for positive and negative target analyte results greater than the absolute value of the IDL for elements not included in the ICSA or ICSAB solutions. Positive results are an indication of either laboratory contamination of the ICS test solutions or an improperly generated interelement correction factor for that element. The latter situation can lead to false positive results on samples.

Aqueous samples that exhibit levels of the four ICSA interferents (Al, Ca, Fe and Mg) at least as high as those present in the ICSAB (500, 500, 200, and 500 milligrams per liter [mg/L], respectively) can be examined for potential false positive and false negative results based on interference correction factor deficiencies. Application of these guidelines to soil samples requires that reduced sample size and dilution effects upon the levels of the four ICSA elements is taken into account. These calculate to be 100,000 milligrams per kilogram (mg/kg) for Al, Ca and Mg, and 40,000 mg/kg for Fe. If the low-level concentrations are used for the ICSA, then the guidelines apply to all recoveries, regardless of the sample concentrations of the interfering elements. The guidelines, which apply, are noted below.

- Check lab data to ensure that the interference check samples were analyzed at the proper frequency.

- Spot check for transcription errors between the ICS recoveries reported on the summary form, and the raw data. Recalculate from the raw data one or more of the analyte percent recoveries and compare to the laboratory reported recovery on the summary form.
- Evaluate the ICS raw data for results with an absolute value greater than the IDL for those analytes that are not present in the ICS solution. If results greater than the IDL are observed an evaluation of the associated sample data for the affected elements should be made. If aluminum (Al), calcium (Ca), iron (Fe), and magnesium (Mg) equals ICS levels, a potential for false positive results exist and professional judgement should be used.

If interference check samples were not analyzed at the proper frequency, use professional judgement to determine the usability of the data.

ICSA/ICSAB CRITERIA AND VALIDATION ACTION

Assessment Element	*Failure*	*Action* [1]		*Reason Code*
		Non-Detects	*Positive Results*	
Accuracy	% Recovery <50% (NFG)	R	J	ICS
	% Recovery >120% (NFG)	NA	J	ICS
	% Recovery 50%-79% (NFG)	UJ	J	ICS
Spectral Interference (non-blank qualified hits in ICSAB)	Positive values	NA	J	ICS
	Negative values (>RL) (NFG)	R	J	ICS

Notes:
1 Action is based on the fact that Al, Ca, Fe, or Mg are present at least as high as ICSAB solution Al, Ca, Mg (500 mg/L or 100,000 mg/kg) and Fe (200 mg/L or 40,000 mg/kg)

6.13 SERIAL DILUTION SAMPLE - INORGANICS

The serial dilution of samples quantitated by ICP and ICP/MS determines whether or not significant physical or chemical interferences exist due to sample matrix. A serial dilution of 1:5 must be performed on at least one sample from every batch of analyses by ICP and ICP/MS to determine if physical or chemical interferences exist in the analyte determinations. Field blanks cannot be used to fulfill the serial dilution requirement. If the analyte in the sample is at least 50 times the value of the IDL, then the percent

difference between the value obtained from the 1:5 dilution and the undiluted value must be within 10 percent. Serial dilution data assessment is completed during FDV only.

- Check lab data to ensure that the serial dilution samples were analyzed at the proper frequency.
- Check the raw data and recalculate the percent difference (%D). Verify that the serial dilution analysis results and the calculated % D agree with the values reported on the summary form.
- Check the raw data for any evidence of negative interference (results from the diluted sample that are significantly higher than the original sample), possibly due to high levels of dissolved solids in the sample, etc.

SERIAL DILUTION CRITERIA AND VALIDATION ACTION

Assessment Element	*Failure*	*Action*		*Reason Code*
		Non-Detects	*Positive Results*	
% Difference	% Difference >10% (Detects >50x IDL) (NFG)	Not Applicable	J (all in batch of similar matrix/concentration)	ISD

6.14 TENTATIVELY IDENTIFIED COMPOUNDS - GC/MS

Tentatively identified compounds (TICs) are only provided upon request and are reviewed during FDV only. TICs are found in the samples that elute from the GC/MS as defined peaks, yet are not calibrated analytes. Up to 30 peaks (10 per VOC and 20 per SVOC fractions) greater than 10 percent in area or height of the nearest internal standard are candidate TICs. The following cautions are appropriate when considering spectral matches from these libraries:

- Library spectra used may not be obtained from mass spectrometers tuned to meet BFB/DFTPP acceptance criteria.
- The source of the compound/spectra in the library may be of questionable integrity (identifications are simply wrong).

- Some of the spectra may have been background corrected to remove artifact signals and others have not.
- In the interest of conservation of storage space, the spectra have been reduced to eliminate many of the small signals, leaving just the major signals.

Check that all TICs include a raw spectra, an enhanced spectra and library matches.

For TICs that are not reported as "unknown":

- Review mass spectra for each TIC identified and compare to the library match selected by the laboratory.
- Ensure that major ions are present in the sample mass spectra.
- Spot-check relative intensities of major ions to verify that they are within 20 percent of the expected values.
- Check to see that all positive identifications have library matches greater than 85 percent.

If there are questions regarding compound identification, contact the laboratory to discuss and obtain corrected forms if needed.

If TICs are identified in blanks at concentrations similar to those in the samples, these TICs should be considered contamination and identified by the laboratory as such.

If any target analytes from another fraction are identified as TICs (e.g., naphthalene in the VOC analysis), check to see that the compound was reported as a target analyte with the proper fraction. If the analyte was reported with the proper fraction, disregard it as a TIC in the other fraction; do not report the analyte in both fractions.

The following TICs are common laboratory artifacts or analytical by-products. If these compounds are not of specific interest to the site, the presence of these compounds should be noted in the data validation memo and they should be disregarded as TICs.

- Common laboratory contaminants: CO_2, siloxanes, diethyl ether, hexane, certain freons, and low level phthalates.

- Solvent preservatives such as cyclohexane and its related by-products (cyclohexanone, cyclohexanol, chlorocyclohexane and chlorocyclohexanol).
- Aldol condensation products (4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-pentene-2-one, and 5,5-dimethyl-2(5H)-furanone).

All TIC data must be qualified as estimated (J). TICs that are identified by the laboratory with a specific compound name, a Chemical Abstracts Service Registry Number (CASRN), and meet the review criteria above are qualified with "NJ", indicating that presumptive evidence of the compound exists and the reported concentration is estimated.

It should be noted that the laboratory may have a more extensive list of calibrated compounds than the requested compound list, and these calibrated compounds may be reported as TICs. Non-target, calibrated compounds reported as TICs are qualified as estimated (J) because they have not been reviewed in the same manner as the target compounds. The data validation report should include a discussion and tabular summary (if appropriate) of these TICs.

6.15 ANALYTE IDENTIFICATION - ORGANICS

Spot check positive sample results to ensure that retention times are within the established retention time windows at a 10 percent frequency.

For GC/MS, spot-check mass spectra at a 10 percent frequency during FDV as follows:

- Perform a visual comparison of sample mass spectra against reference spectra to ensure a general match.
- Verify that all major ions are present and their intensities are within 20 percent of the expected values.
- Review ions in the sample with >10 percent abundance that are not in the reference spectra.

If there are any discrepancies in analyte identification, the laboratory must be contacted for resolution and corrected forms submitted if necessary. Qualification of data due to analyte identification discrepancies are summarized below.

ANALYTE IDENTIFICATION APPROVAL CODES

Assessment Element	*Failure*	*Action*		*Reason Code*
		Non-Detects	*Positive Results*	
Second Column confirmation (1)	%D > 40% ≤80% %D >80%	None None	J NJ	DCD DCD
GC Degradation	> 15 %for GC	None	J	DEG
Chromatography	Poor chromatography	Prof. Judgement	Prof. Judgement	QUA
Carry over contamination	Suspected instrumental carryover	Prof. Judgement	Prof. Judgement	SIC

Notes: (1) If reported result <RL and %D >50, qualify as non-detect at the RL.

6.16 ANALYTE QUANTITATION

Analyte quantitation is verified at a 10 percent frequency during FDV with the following checks:

- Spot check sample report limits for non-detected analytes (for organics and general chemistry analytes, use the lowest calibration standard to verify the quantitation limit; for metals, use the IDL to verify the report limit), making sure that report limits reflect individual original sample weights/volumes, final volumes, percent moisture (where applicable), dilutions, etc.
- Spot-check random positive sample results.
- Note in the data validation text whether values below the report limit were reported and qualified as estimated (lab "J" or "B" flagged values). These analytes are qualified as J values with the BRL reason code.
- If the project requires detection limit reporting (sample data are reported non-detect to the laboratory sample specific MDLs) all non-detect data should be qualified as estimated values (UJ) with the DLR reason code.

- Review replicate or quadruplicate results as specified in methods (graphite furnace, total organic carbon [TOC], and total organic halides [TOX]).
- Note in the data validation text whether soil results were reported on a dry- or wet-weight basis.
- If dilutions were performed, ensure that values above the calibration range (E values) are not used unless absolutely necessary (these values should be qualified as estimated). Compare original and diluted results to ensure that they are compared.

If there are any discrepancies in analyte quantitation, the laboratory must be contacted for resolution and corrected forms submitted if necessary. Qualification of data based on analyte quantitation discrepancies observed are summarized below.

ANALYTE QUANTITATION APPROVAL CODES

Assessment Element	*Failure*	*Action*		*Reason Code*
		Non-Detects	*Positive Results*	
Exceeds Calibration Range	Sample results exceeds instrument calibration range	None	J	EXE
Method of Standard Addition	Percent recovery outside method criteria	UJ	J	MSA
Variability	Variability in replicate results	Prof. Judgement	Prof. Judgement	REP
Precision-Metals	%RSD outside criteria for replicate aspiration	Prof. Judgement	Prof. Judgement	RSP
Percent Solids	Percent solids less than 50%	Prof. Judgement	Prof. Judgement	SLD
Variability in Sample Results	Dissolved analyte result significantly greater than total	Prof. Judgement	Prof. Judgement	TVD
Dilutions	Validator's choice of dilution	Prof. Judgement	Prof. Judgement	VCD
Reanalysis	Validator's choice of reanalysis	Prof. Judgement	Prof. Judgement	VCR

6.17 FIELD DUPLICATES

Overall precision for the sampling event and laboratory procedures is monitored through the collection and analysis of field duplicate sample sets.

- Compare results for original sample and field duplicate and calculate the RPD. If one or more results are non-detect, use the report limit or MDL value to calculate the RPD.
- Compare the RPDs to the project control limits. If limits have not been established, use default limits of 50 percent for water and 100 percent for soil samples if the sample results are greater than five times the RL. If sample results are less than 5 times the RL, precision is assessed by comparing the difference between the two results to a control limit of plus or minus one time the RL value for water samples and two time the RL value for soil samples.

If outlying RPDs are identified, qualify all associated positive results for that sample and it's duplicate as summarized below. If the field duplicate results show drastic differences, check with the sampler to ensure proper field duplicate identification, then have the laboratory verify proper sample labeling and analyte quantitation.

FIELD DUPLICATE SAMPLE ASSESSMENT

Assessment Element	*Failure*	*Action*		*Reason Code*
		Non-Detects	*Sample Detections*	
Precision				
Sample or duplicate concentrations > 5X RL	Water sample RPD > 50%	None (CRA)	J (CRA)	FDP
	Soil sample RPD > 100%	None (CRA)	J (CRA)	FDP
Sample or duplicate concentrations < 5X RL	Water sample difference >1X RL	None (CRA)	J (CRA)	FDP
	Soil sample difference >2X RL	None (CRA)	J (CRA)	FDP

7.0 LAB RESUBMISSION DOCUMENTATION

Corrections to laboratory reports must be requested in writing and a hardcopy of this request must be stored with the project files. Obsolete sample pages must be stamped "Corrected" and signed and dated by the data validator. The corrected pages are to be inserted in the original data package.

If a complete package resubmission is requested, the obsolete package must be marked "obsolete" and the new package include a summary of the changes in the report narrative.

8.0 FLAT FILE CHECKS

Prior to extracting an analytical table from the flatfile, the following checks must be performed:

i) ensure for each sample that there is only one result designated "reportable" for all associated analyte;

ii) verify that acceptance codes for all results reflect the level of data review performed by the validator;

iii) ensure that all lab qualifiers have been addressed and converted if necessary to data validation qualifiers (e.g., B, E, *, etc.).

iv) ensure that all validation qualifications have appropriate Reason Codes

Ensure that all results qualified "U" or "UJ" have "N" in the detected field.

9.0 REPORT FORMAT

Data validation report should include the following:

i) an introduction detailing the objective of the sampling and analysis program; general sample collection information; the analytical methods used; and a reference of all pertinent quality documents;

ii) text describing the elements of the data package that were reviewed, the findings for each, and the impact each finding had on the overall data usability;

iii) a sampling and analysis summary table; and

iv) tables summarizing all data qualification and the rationale for each.

An example data validation report (FDV) is presented in Appendix B.

10.0 REFERENCES

- "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", USEPA-540/R-99/008, October 1999
- "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Review", USEPA-540/R-94/013, February 1994.
- "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Review", USEPA-540/R-01/008, July 2002.
- "Region III Modifications to National Functional Guidelines for Organic Data Review", USEPA, September 1994.
- "Region 5 Standard Operating Procedure for Validation of CLP Organic Data", USEPA February 1997.
- "Quality Assurance/Quality Control Guidance for Removal Activities, Sampling QA/QC Plan and Data Validation Procedures", USEPA/540/G-90/004, OSWER Directive 9360.4-01, dated April 1990.
- USEPA Guidance on Environmental Data Verification and Data Validation, USEPA QA/GA-8, November 2002.
- "Quality Assurance Associates Standard Operating Procedure for the Data Quality Review (DQR) of Organic and Inorganic Data", October 1999.
- "USEPA Guidance for Quality Assurance Project Plans", December 2002, USEPA/240/R-02/009.
- Environmental Protection Agency, Region III. Innovative Approaches to Data Validation. 1995. U.S. Environmental Protection Agency, Region III. USEPA Region III QA Directives. U.S.

11.0 GLOSSARY OF TERMS

Accuracy	A measure of overall agreement of a measurement to a known value. Accuracy is assessed by means of percent recoveries and reference samples.
Assessment	The evaluation process used to measure the performance or effectiveness of a system and its elements.
Action Level	The concentration level that is high enough to warrant action. Action levels are generally regulatory levels that are determined by a regulatory agency.
Analyte	The element or compound to be determined.
Blank	A purified sample matrix subjected to the usual analytical or measurement process. A blank should not contain analytes of interest. A blank is used to detect contamination during sampling, handling, preparation, and/or analysis.
Calibration Curve	The relationship between instrument response and analyte concentration. The "curve" may be linear or non-linear.
Calibration Factor	The response factor for external standard analyses expressed as peak area (or height) per nanogram of analyte injected.
Calibration Range	The concentration range bounded by the lowest and highest concentration calibration standards used in the equation for the calibration curve.
Continuing Calibration	The daily standard when it is used to update the response factors for sample quantitation.
Chain of Custody	An unbroken trail of accountability that ensures the physical security of samples, data, and records.
Comparability	A measure of the confidence with which one data set or method can be compared to another.
Completeness	A measure of the amount of valid data obtained from a measurement system.
Compliance Review	Compliance review is the assessment of data package completeness and compliance with project requirements as outlined in the project Analytical Scope of Work.
Daily Standard	A single calibration standard, normally at a concentration near the middle of the calibration range, that is analyzed at the beginning of each analysis shift.

Data Quality	A measure of the degree of acceptability or utility of data for a particular purpose.
Data Validation	Data validation is an analyte-specific and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance to determine the analytical quality of a specific data set.
Data Validation-Reduced or Data Verification	Reduced Data Validation (RDV) is assessment of data and quality control deliverables provided in a standard summary laboratory report. Qualification of sample results is based on sample holding time periods, sample preservation, and laboratory batch quality control sample results (method blank, laboratory control samples, matrix spike/matrix spike duplicate or matrix spike/matrix duplicate, and surrogates) in accordance with U.S. USEPA's National Functional Guidelines, the analytical methods, and project quality assurance documents.
Data Validation-Full	Full Data Validation (FDV) is assessment of data and quality control deliverables provided in an expanded laboratory report . Qualification of sample results is based on sample holding time periods, sample preservation, laboratory batch quality control sample results (method blank, laboratory control samples, matrix spike/matrix spike duplicate or matrix spike/matrix duplicate, and surrogates), instrument calibration data, analyte identification and quantitation, additional method-specific quality control results (as appropriate), and raw data in accordance with U.S. USEPA's National Functional Guidelines, the analytical methods, and project quality assurance documents. FDV consists of reviewing and assessing all sample results and quality control data reported in summary tables and a 10 percent spot check of laboratory calculations and analyte identification from the raw data.
Field Duplicate	A second aliquot of a sample collected in the field that is treated the same as the original in order to determine the precision of the sampling and analysis.
Field Blank (Trip, Rinsate)	A blank that measures contamination possibly introduced in the field or during transportation. It also includes any contamination introduced during sample handling, preparation and/or analysis.

Initial Calibration Verification	The daily standard when it is used to verify that the calibration curve is still valid for sample quantitation (see also continuing calibration standard).
Inorganics	Inorganics as specified within this document are metals and cyanide.
Instrument Detection Limit	The minimum concentration of a target analyte, determined by various means, that can be measured above the instrument background noise.
Laboratory Control Sample	An aliquot of a clean matrix, spiked with known quantities of selected analytes.
Matrix	The predominant material of which the sample is composed. Matrix is not a synonymous phase.
Matrix Duplicate	A second aliquot of a sample that is treated the same as the original in order to determine the precision of the method.
Matrix Spike/Spike Duplicate	An aliquot of a sample spiked in duplicate with known quantities of selected analytes.
Method	A body of procedures and techniques for performing an activity, systematically presented in the order in which they are to be executed.
Method Blank	A blank prepared to represent the sample matrix as closely as possible and analyzed exactly like the calibration standards, samples, and QC samples. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the analytical procedure.
Method Detection Limit	The statistically derived lowest level of an analyte in a sample that will result in a signal different than zero as specified in 40 CFR 136, Appendix B.
Organics	Organics as specified within this document are volatile organic compounds, semi-volatile organic compounds, and polychlorinated biphenyls.
Parent Sample	The sample from which an aliquot was taken by the laboratory to prepare a matrix spike or analyzed as a matrix duplicate. This also refers to the investigative sample and field duplicate relationship.
Percent (%) Difference	A parameter used to compare the daily standard to the initial calibration when a calibration or response factor is

	used, comparing the difference of the factors. It indicates both the direction and the magnitude of the comparison.
Percent (%) Drift	A parameter used to compare the daily standard to the initial calibration when a linear calibration curve is used, comparing the % difference of the determined concentrations. It indicates both the direction and the magnitude of the comparison.
Precision	A measure of agreement among repeated measurements of the same property under identical, of substantially similar, conditions. Precision is assessed by means of duplicate/replicate sample analysis.
Quality Assurance	A system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Control	A set of measures and activities that are used to fulfill the need for quality.
QC Sample	An uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.
Quantitation	The degree to which the instrument measures the concentration of target analytes.
Quantitation Limit	The minimum concentration that a target analyte can be measured within specified limits of precision and accuracy. Quantitation limits are generally 5-10x the method detection limit.
Re-analysis	The process of repeating a sample that includes both a new sample preparation and analysis.
Recovery	The act of determining whether or not the methodology measures all of the analyte contained in a sample.
Re-injection	The process of repeating a sample that does not include a new sample preparation. It is a re-injection of an existing extract.
Reported Result	The concentration or a target analyte in a given sample that is reported by the laboratory on the analysis results form. A

	positive result that is below the report limit may or may not be a reported result depending on the client's request.
Report Limit	The detection limit or quantitation limit (depending on the client's request) for a target analyte in a given sample that is reported by the laboratory on the analysis results form. The value includes adjustment for any sample dilution or method factor.
Representativeness	The measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.
Sample	An environmental sample that is not a QC sample and any re-analysis or re-injection thereof.
Sensitivity	The capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest.
Spike	A substance that is added to an environmental sample to increase the concentration of the target analyte by a known amount. A laboratory control spike or matrix spike is used to measure accuracy. A spike duplicate is used to measure precision.
Split Samples	Two or more representative portions taken from one sample in the field or in the laboratory and analyzed by different analysts or laboratories. Split samples are quality control samples that are used to assess analytical variability and comparability.
Standard Operating Procedure	A document that details the method for an operation, analysis, or action which thoroughly describes the techniques and steps to be followed. It is officially approved as the method for performing certain routine or repetitive tasks.
Target Analyte	An analyte specifically reported for a given analysis.

TABLE 2.1

DATA REVIEW AND VALIDATION LEVELS

Item Reviewed	*Compliance Review*	*Reduced Data Validation*	*Innovative Validation*	*Full Data Validation*
General Report Deliverables				
Sample ID Check (COC versus Lab Deliverables)	X	X	X	X
Methods/Procedures	X	X	X	X
Parameter List	X	X	X	X
Report/Detection Limits	X	X	X	X
Documentation/Deliverables	X	X	X	X
Sample Specific and Batch QC Results				
Sample Preservation and Holding Times		X	X	X
Method Blanks		X	X	X[1]
Field Blanks (Trip and Rinsate Blanks)		X	X	X[1]
System Monitoring Compounds (Surrogates)		X	X	X[1]
MS/MSD - Organics		X	X	X[1]
MS/MSD, MS/MD - Inorganics		X	X	X[1]
Laboratory Control Sample (LCS)		X	X	X[1]
Field Duplicates		X	X	X[1]
Expanded Data Elements				
Instrument Performance Check (GC/MS & ICP/MS)			X	X[1]
Initial Calibration - Organics			X	X[1]
Continuing Calibration - Organics			X	X[1]
Initial Calibration Verification - Inorganics			X	X[1]
Continuing Calibration Verification - Inorganics			X	X[1]
Internal Standards (GC/MS & ICP/MS)			X	X[1]
Instrument Blanks - Inorganics			X	X[1]
ICP/MS Internal Standards			X	X[1]
ICP Interference Check Samples			X	X[1]
Serial Dilutions			X	X[1]
Compound Identification				X[1]
Chromatography				X[1]
Compound/Analyte Quantitation (raw data)				X[1]
Report Limit Verification				X[1]

Notes:

[1] Raw data review including calculation checks and chromatography review will be completed on 10 percent of the sample data unless data warrants otherwise.

GC Gas Chromatograph.

ICP Inductively Coupled Plasma.

MS Matrix Spike.

MSD Matrix Spike Duplicate.

PROPRIETARY DOCUMENT

TABLE 3.1

DATA VALIDATION LEVELS - "APPROVAL_CODES"

Approval Code	*Description*
0	Validation Status Unknown
1	No Review Performed
2	Reduced Validation/Verification Performed
3	Full Validation Performed
4	Lab Qualified Result, in the process of Verification/Validation
5	Do not use
6	Compliance Check
8	Full Validation Performed by External Consultant
9	Reduced Validation/Verification Performed by External Consultant
10	Innovative Approach Validation (Forms Review includes NJ Reduced)

TABLE 4.1

DATA VALIDATION GUIDANCE DOCUMENTS - "APPROVAL_b"

Guidance Code[1]	*Data Validation Guidance Document(s)*
01	National Functional Guidelines in conjunction with CRA SOP
02	CRA Analytical Data Quality Assessment and Validation SOP 2004
03	NJDEP Guidance Documents in conjunction with CRA SOP
04	Region II Guidance Documents in conjunction with CRA SOP
05	Region III Guidance Documents in conjunction with CRA SOP
06	Region I Guidance Documents in conjunction with CRA SOP
07	Region 5 Guidance Documents in conjunction with CRA SOP
08	Texas Commission on Environmental Quality) Regulatory Guidance Texas Risk Reduction Program (TRRP-13) in
09	Canadian Validation Procedural Review
10	Guidance Document Unavailable (professional judgement used for data validation)
11	National Functional Guidelines in conjunction with CRA SOP and QAPP
97	Unknown DV Guidance Document
98	Not applicable. Data not validated
99	Predates population of approval_b field as of October 26, 2005

Note:

1 Guidance codes are identified as Approval_b codes in the database and flat files

CRA Conestoga-Rovers & Associates.

DV Data Validation.

NJDEP New Jersey Department of Environmental Protection.

SOP Standard Operating Procedure.

TABLE 5.1

DATA VALIDATION REASON CODES - "APPROVAL_a"

Reasons for Data Qualification	*Reason Code*
Analyte present, quantitation may be biased high due to presence of co-eluting target analyte(s).	ABH
Result reported is below calibration level; quantitation may not be accurate.	BCL
Breakthrough of Aromatic Compounds into the Aliphatic Fraction.	BKR
Below Reporting Limit (for preserving laboratory organic Js and inorganic Bs)	BRL
Qualified due to blank spike outlier	BSQ
Inorganics calibration blank contamination - initial or continuing	CBK
Continuing Calibration Outlier	CCL
CRA/Contract Required Detection Limit (CRDL) Outlier	CRA
Dual column discrepancy (for GC analyses only)	DCD
Qualified due to high degradation (GC pest analysis only)	DEG
Detection Limit Reporting	DLR
Duplicate Variability	DUP
Exceeds calibration range	EXE
Field Blank Contamination	FBK
Field Duplicate Variability	FDP
Filter Blank Contamination	FIL
Holding Time Exceedance	HTQ
Dioxin/Furan Ion Abundance Ratio Violation	IBA
Initial Calibration Outlier	ICL
Inductively Coupled Plasma (ICP) Calibration Stability (instrument drift)	ICS
ICP Serial Dilution	ISD
ICP Interference Check Sample Outlier	ISI
Internal Standard Outlier	IST
Laboratory Control Sample (LCS) Percent Recovery Outlier	LCQ
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCD) Outlier	LCD
Method Blank Contamination	MBK
Method of Standard Additions Outlier (Metals)	MSA
Matrix Spike/Matrix Spike Duplicate (MS/MSD) Outlier	MSD
Matrix Spike (MS) Percent Recovery Outlier	MSQ
Nitrite results greater than combined nitrate/nitrite result	NNQ
Preservation of laboratory qualifier	PLQ
Preservation of laboratory U qualifier	PLU
Post Digestion Spike Outlier	PSP
Analyte present, the reported value may not be accurate or precise due to poor chromatography. The sample chromatogram exhibits baseline interference that impacted sample quantitation.	QUA
Rinse Blank Contamination	RBK
Variability in replicate results	REP
Percent Relative Standard Deviations (% RSDs) out for replicate aspiration	RSP
Suspected Instrumental Carryover	SIC
Percent Solids less than 50%	SLD
Sample preservation violation due to - temperature, preservative and pH	SPV
Sample Receipt Nonconformance	SRN
Surrogate Outlier	SUR
Trip Blank Contamination	TBK

TABLE 5.1

DATA VALIDATION REASON CODES - "APPROVAL_a"

Reasons for Data Qualification	*Reason Code*
Tentatively Identified Compound (TIC)	TIC
Total dioxins and/or furans are reported as estimated due to the nature of the quantitation.	TOT
Tune Nonconformance	TUN
Dissolved analyte result is significantly greater than the associated total analyte result	TVD
Validator's Choice of Columns for reporting GC results	VCC
Validator's choice of dilutions for when the dilution on the flat file is not the one you want to report	VCD
Validator's choice of method	VCM
Validator's choice of reanalysis	VCR

TABLE 6.1
DATA VALIDATION QUALIFIER DEFINITION

Qualifier[1]	*Definition*
Organics	
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample (for detected
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification".
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely
Inorganics	
U	The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
J	The associated value is an estimated quantity.
R	The data are unusable. (Note: Analyte may or may not be present).
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise

Notes:

1 Qualifier codes identified are based on the following guidance documents.

Organics "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, USEPA 540/R-99/008, October 1999.

Inorganics "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, USEPA 540/R-94/013, February 1994.

TABLE 6.2

ABBREVIATION OF DATA VALIDATION QUALIFIER DEFINITIONS

Qualifier[1]	*Definition*
	Organics & Inorganics
J	Estimated Concentration
N	TIC Presumptively Present
NJ	TIC Presumptively Present, Estimated Concentration
R	Rejected
U	Not Detected
UJ	Not Detected, Estimated Reporting Limit

Notes:

[1] Qualifier codes identified are based on the following guidance documents.

Organics "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, USEPA 540/R-99/008, October 1999.

Inorganics "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, USEPA 540/R-94/013, February 1994.

TIC Tentatively Identified Compound.

TABLE 6.3

SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2] *40 CFR*	*SW-846*	*Volume of Sample*
WATER *Organic Tests*					
VOC	Three 40 mL teflon-lined septum vials per analysis	HCl to pH < 2 Iced, 4 ± 2° C	14 days for analysis	14 days for analysis	Fill completely, no air bubbles
VOC-unpreserved	Three 40 mL teflon-lined septum vials per analysis	Iced, 4 ± 2° C	7 days for analysis	7 days for analysis	Fill completely, no air bubbles
PCB as Aroclors	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	1 year until extraction, 1 year after extraction	None	Fill to neck of bottle
Polynuclear Aromatic Hydrocarbons (PAH) or Base-Neutral/Acid Extractables (BNA)	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	7 days for extraction 40 days after extraction for analysis	7 days for extraction 40 days after extraction for analysis	Fill to neck of bottle
Pesticides	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	7 days for extraction 40 days after extraction for analysis	7 days for extraction 40 days after extraction for analysis	Fill to neck of bottle
Herbicides	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	7 days for extraction 40 days after extraction for analysis	7 days for extraction 40 days after extraction for analysis	Fill to neck of bottle
PCDD/PCDF (Dioxins/Furans)	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	1 year	None	Fill to shoulder of jar
Formaldehyde	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	-	3 days for extraction; 3 days from extraction to analysis	

TABLE 6.3

SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2] *40 CFR*	*SW-846*	*Volume of Sample*
Organic Tests (Cont'd.)					
TPH as Gas Range	Three 40 mL teflon-lined septum vials per analysis	Iced, 4 ± 2° C	-	14 days for analysis	Fill completely, no air bubbles
TPH as Diesel Range	Two 1 liter amber glass bottles per analysis	HCl to pH < 2 Iced, 4 ± 2° C	-	7 days for extraction 40 days after extraction for analysis	Fill to neck of bottle
Inorganic Tests					
Acidity	One 250 ml plastic bottle	Iced, 4 ± 2° C	14 days for analysis	-	Fill to neck of
Alkalinity	One 250 ml plastic bottle	Iced, 4 ± 2° C	14 days for analysis	-	Fill to neck of bottle
Ammonia, nitrogen	One 500 ml plastic bottle	H_2SO_4 to pH < 2 Iced, 4 ± 2° C	28 days for analysis	-	Fill to neck of bottle
Biochemical Oxygen Demand (BOD)	One 1 liter amber glass bottle	Iced, 4 ± 2° C	48 hours to initiate analysis	-	Fill to neck of bottle
Bromide	One 250 ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	28 days for analysis	Fill to neck of bottle
Chloride	One 250 ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	28 days for analysis	Fill to neck of bottle
Chemical Oxygen Demand (COD)	One 250 ml plastic bottle	H_2SO_4 to pH < 2 Iced, 4 ± 2° C	28 days for analysis	-	Fill to neck of bottle
Hexavalent Chromium	One 250 ml plastic bottle	Iced, 4 ± 2° C pH=9.3-9.7, cool ≤6°C	24 hours for analysis 28 days for analysis	-	Fill to neck of bottle

TABLE 6.3
SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2]		*Volume of Sample*
			40 CFR	*SW-846*	
Inorganic Tests (Cont'd.)					
Coliform, Total/Fecal	One 250 ml plastic bottle (sterile)	Iced, 4 ± 2° C	6 hours for analysis	6 hours for analysis	Fill to neck of bottle
Color	One 250 ml plastic bottle	Iced, 4 ± 2° C	48 hours for analysis	-	Fill to neck of bottle
Cyanide (total, free or amenable)	One 250 ml plastic bottle	NaOH to pH>12 Iced, 4 ± 2° C	14 days for analysis	14 days for analysis	Fill to neck of bottle
Fluoride	One 250 ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	28 days for analysis	Fill to neck of bottle
Hardness	One 250 ml plastic bottle	HNO_3 to pH < 2 Iced, 4 ± 2° C	6 months	6 months	Fill to neck of bottle
Metals	One 1 liter plastic bottle	HNO_3 to pH < 2 Iced, 4 ± 2° C	6 months	6 months	Fill to neck of bottle
Mercury	One 1 liter plastic bottle	5ml/L 12N HCl or BrCl	90 days	28 days for analysis	
Low Level Mercury[3]	Four 40 mL teflon-lined septum vials per analysis	Iced, 4 ± 2° C	-	48 hours until laboratory preservation with BrCl, 28 days from collection until analysis	Fill completely, no air bubbles
Nitrogen, Kjeldahl (TKN)	One 250 ml plastic bottle	H_2SO_4 to pH < 2 Iced, 4 ± 2° C	28 days for analysis	-	Fill to neck of bottle
Nitrate or Nitrite	One 250 ml plastic bottle	Iced, 4 ± 2° C	48 hours for analysis	48 hours for analysis	Fill to neck of bottle

TABLE 6.3

SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2] *40 CFR*	*SW-846*	*Volume of Sample*
Inorganic Tests (Cont'd.)					
Nitrate plus Nitrite	One 250 ml plastic bottle	H_2SO_4 to pH < 2 Iced, 4 ± 2° C	28 days for analysis	28 days for analysis	Fill to neck of bottle
Oil & Grease	One 1 liter amber glass bottle	H2SO4 or HCl to pH<2 Iced, 4 ± 2° C	28 days for analysis	28 days for analysis	Fill to neck of bottle
Phenols	One 500 ml glass bottle	H_2SO_4 to pH < 2 Iced, 4 ± 2° C	28 days for analysis	28 days for analysis	Fill to neck of bottle
Phosphate	One 250 ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	48 hours for analysis	Fill to neck of bottle
Phosphorus (total)	One 500 ml plastic bottle	H_2SO_4 to pH < 2 Iced, 4 ± 2° C	28 days for analysis	-	Fill to neck of bottle
ortho-Phosphate	One 250 ml plastic bottle	Iced, 4 ± 2° C	48 hours for analysis	-	Fill to neck of bottle
Paint Filter	One 500 ml glass bottle	Iced, 4 ± 2° C	-	None	Fill to neck of bottle
pH	One 250 ml plastic bottle	Iced, 4 ± 2° C	15 minutes	-	Fill to neck of bottle
Radiochemistry:					
Alpha, Beta, Radium	Two 1 liter plastic bottles	HNO_3 to pH < 2, Iced, 4 ± 2° C	6 months for analysis	-	Fill to neck of bottle
Tritium	One 250 ml glass amber bottle	Iced, 4 ± 2° C	6 months for analysis	-	Fill to neck of bottle
Radon	Three 40 mL glass vials	Iced, 4 ± 2° C	4 days for analysis	-	Fill to neck of bottle
I-131	One 1 liter plastic bottle	NaOH to pH>8 Iced, 4 ± 2° C	16 days for analysis	-	Fill to neck of bottle

TABLE 6.3

SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2] *40 CFR*	*SW-846*	*Volume of Sample*
Inorganic Tests (Cont'd.)					
Silica	One 250 ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	-	Fill to neck of bottle
Solids Total Dissolved Solids (TDS) Total Solids(TS) Total Suspended Solids (TSS) Suspended Solids (SS)	One 250 ml plastic bottle	Iced, 4 ± 2° C	7 days for analysis	-	Fill to neck of bottle
Sulfide, Total or Reactive	One 500 ml plastic bottle	Zn Acetate/NaOH, pH>9 Iced, 4 ± 2° C	7 days for analysis	7 days for analysis	Fill to neck of bottle
Sulfate	One 250 ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	28 days for analysis	Fill to neck of bottle
Sulfite	One 250 ml plastic bottle	Iced, 4 ± 2° C	15 minutes	-	Fill to neck of bottle
Surfactants	One 250 ml plastic bottle	cool, ≤6°C	48 hours for analysis	-	
Specific Conductance	One 250 ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	-	Fill to neck of bottle
Turbidity	One 250 ml plastic bottle	Iced, 4 ± 2° C	48 hours for analysis	-	Fill to neck of bottle
Total Organic Carbon (TOC)	One 500 ml plastic bottle	H_2SO_4 to pH < 2 Iced, 4 ± 2° C	28 days for analysis	28 days for analysis	Fill to neck of bottle
Total Organic Halogens (TOX)	One 500 ml amber glass bottle	H_2SO_4 to pH < 2 Iced, 4 ± 2° C	-	28 days for analysis	Fill to neck of bottle
Recoverable	One 1 liter amber	HCl to pH < 2	28 days for analysis	-	Fill to neck of
Total Petroleum Hydrocarbons (TPH)	glass bottle	Iced, 4 ± 2° C			bottle

TABLE 6.3
SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2] *40 CFR*	*SW-846*	*Volume of Sample*
SOLID (Soil/Sediment)					
VOC[4,5]	One 25g En Core Sampler™ per analysis or field-preserve w/ methanol	Iced, 4 ± 2° C	-	48 hours for extraction(En Core 14 days for analysis	Fill completely
PCB as Aroclors	One 4-ounce glass jar	Iced, 4 ± 2° C	-	none	Fill to shoulder of jar
Pesticides	One 4-ounce glass jar	Iced, 4 ± 2° C	-	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar
Polynuclear Aromatic Hydrocarbons (PAH) or Base-Neutral/Acid Extractables (BNA)	One 4-ounce glass jar	Iced, 4 ± 2° C	-	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar
Herbicides	One 4-ounce glass jar	Iced, 4 ± 2° C	-	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar
PCDD/PCDF (Dioxins/Furans)	One 4-ounce glass jar	Iced, 4 ± 2° C	-	none	Fill to shoulder of jar
Metals	One 4-ounce glass jar	Iced, 4 ± 2° C	-	180 days (mercury 28 days) for analysis	Fill to shoulder of jar
Cyanide (total)	One 4-ounce glass jar	Iced, 4 ± 2° C	-	14 days for analysis	Fill to shoulder of jar
Hexavalent Chromium	One 4-ounce glass jar	Iced, 4 ± 2° C	-	30 days for extraction 168 hours for analysis	Fill to shoulder of jar

TABLE 6.3
SAMPLE PRESERVATION AND HOLDING TIME REQUIREMENTS

Analyses	*Sample Containers*[1]	*Preservation*	*Maximum Holding Time from Sample Collection*[2]		*Volume of Sample*
			40 CFR	*SW-846*	
TCLP					
VOC	One 4-ounce glass jar	Iced, 4 ± 2° C	-	14 days from collection to TCLP extraction; 14 days from extraction to analysis	Fill to shoulder of jar
SVOC	One 4-ounce glass jar	Iced, 4 ± 2° C	-	14 days from collection to TCLP extraction; 7 days from TCLP extraction to prep extraction; 40 days from prep extraction to analysis	Fill to shoulder of jar
Metals	One 4-ounce glass jar	Iced, 4 ± 2° C	-	180 days from collection to TCLP extraction; 180 days from extraction to analysis	Fill to shoulder of jar
Mercury	One 4-ounce glass jar	Iced, 4 ± 2° C	-	28 days from collection to TCLP extraction; 28 days from extraction to analysis	Fill to shoulder of jar

Notes:
[1] - Multiple parameters on a single sample with identical preservation requirements may be combined into one single sample container.
[2] - These are technical holding times, i.e., are based on time elapsed from time of sample collection.
[3] - Sample containers must be fluoropolymer or borosilicate glass.
[4] - If Encore™ samples cannot be analyzed within 48 hours, they can be frozen at -10 degrees Celcius.
[5] - If no other samples are submitted with solid VOCs a separate container must be included for percent moisture.

APPENDIX A-1

LABORATORY REPORT DELIVERABLES CHECKLIST – QC SUMMARY REPORT

APPENDIX A-1

LABORATORY REPORT DELIVERABLES CHECKLIST - ORGANIC DATA

Case Narrative:

	Case Narrative Present

Quality Control Summary Package:

	Data Summary Sheets
	Matrix Spike/Spike Duplicate Recoveries
	Laboratory Control Sample Recoveries
	Method Blank Summaries
	GC/MS Tuning and Mass Calibration
	Initial Calibration Data
	Continuing Calibration Data
	Surrogate Compound Recovery Summary
	Internal Standard Area Summary

Sample and Blank Data Package Section

	Reconstructed Ion Current (RIC) Chromatogram
	Quantitation Reports
	Raw and Enhanced Mass Spectra
	Reference Mass Spectra for Target Compounds
	Mass Spectral Library Search for TICs

Raw QC Data Package Section

	DFTPP and/or BFB mass spectra and mass listings
	RIC Chromatogram for Standards, LCS, and MS/MSD
	Quantitation Reports for Standards, LCS, and MS/MSD
	List of Instrument Detection Limits
	Chain of Custody Records
	Sample Preparation and Analysis Run Logs

APPENDIX A-1

LABORATORY REPORT DELIVERABLES CHECKLIST - INORGANIC DATA

Case Narrative:

	Case Narrative present

Quality Control Summary Package:

	Data Summary sheets
	Initial and Continuing Calibration results
	CRDL Standard results
	Preparation Blank and Calibration Blank results
	ICP Interference Check Sample results
	Matrix Spike recoveries
	Matrix Duplicate results
	Laboratory Control Sample recoveries
	Method of Standard Additions results
	ICP Serial Dilution results
	Instrument Detection Limits
	ICP Interelement Correction Factors
	ICP Linear Ranges
	Preparation Log
	Analysis Run Log

Raw QC Data Package Section

	Chain of Custody Records
	Instrument Printouts
	Sample Preparation Notebook Pages
	Logbook and Worksheet Pages
	Percent Solids Determination

APPENDIX A-2

LABORATORY REPORT DELIVERABLES CHECKLIST – EXPANDED DELIVERABLE REPORT

APPENDIX A-2
LABORATORY REPORT DELIVERABLES CHECKLIST
EXPANDED DATA REPORT

Laboratory Name: _____

Date and Report No.: _____

	Report Item	*Complete Yes/No*	*Comments*
A. REPORT COVER PAGE			
1.	CRA Project reference number.		
2.	CRA Project name.		
3.	CRA Work Order representative.		
4.	Purchase Order number.		
5.	Laboratory Short Form representative - Laboratory Project Manager.		
6.	Approval signature.		
7.	Report date.		
B. PROJECT SUMMARY/NARRATIVE			
1.	Summarize project and all Quality Control compliance issues.		
C. SUPPORTING DOCUMENTATION			
1.	employed for the analyses.		
2.	identification numbers.		
3.	Qualifying codes glossary - Define any qualifying codes utilized in report.		
D. TABULATED DATA			
	The sample result section of the report shall include the following information for each sample (one		
1.	CRA sample identification number.		
2.	Laboratory sample identification number.		
3.	Sample collection date and time.		
4.	Dates - Sample collected, Sample Receipt, prepared and analyzed.		
5.	Sample matrix.		
6.	Parameter units of measure.		
7.	Specific analytical methodology.		
8.	QC batch identifier number.		
9.	Percent dry weight (solid samples).		
10.	Dilution factors.		
11.	Test parameters or analytes.		
12.	Analytical results, original and reanalysis due to QC criteria failure or dilutions.		
13.	Sample specific reporting limits.		
14.	Qualifying codes, as required.		

APPENDIX A-2
LABORATORY REPORT DELIVERABLES CHECKLIST
EXPANDED DATA REPORT

Laboratory Name: _____
Date and Report No.: _____

	Report Item	*Complete Yes/No*	*Comments*
E. QUALITY CONTROL NARRATIVE			
	For each sample for which QA/QC problems are encountered, the following specific information shall be		
1.	CRA and Laboratory sample identification numbers affected.		
2.	Sample matrix.		
3.	Parameters affected.		
4.	Data acceptance criteria exceeded.		
5.	Specific analytical problems that occurred.		
6.	Corrective actions(s) taken, or attempted, to resolve the problems.		
F. QUALITY CONTROL RESULTS SUMMARY			
1.	Laboratory method blank results.		
2.	Laboratory control sample or QC check sample - Percent recoveries and acceptance		
3.	Matrix Spike/Matrix Spike Duplicate or Matrix Spike/Matrix Duplicate - Amount in		
4.	Surrogate compounds - Percent recoveries and laboratory control limits (may be included		
5.	QC batch identification numbers.		
G. GC/MS DATA			
1.	Initial and continuing calibration results and summary, including compound relative		
2.	Tuning results and summary report.		
3.	Internal standard response and retention times summary.		
4.	Sample preparation documentation.		
5.	Instrument run logs.		
6.	Labeled and dated chromatograms/spectra of sample results, all laboratory		
7.	Results of tentatively identified compounds, if requested.		
H. GC DATA			
1.	Initial and continuing calibration results and summary, including compound response		
2.	Chlorinated pesticide breakdown report (if applicable).		
3.	Sample preparation documentation.		
4.	Instrument run logs.		
5.	Labeled and dated chromatograms of samples and all laboratory calibration/QC checks.		
I. ICP DATA			
1.	Initial and continuing calibration standard and blank results and summary.		
2.	Low level check standard (CRQL) results and summary.		
3.	Interference check sample results and summary.		
4.	Serial dilutions and post digestion spike results and summary		
5.	Sample preparation records.		
6.	Instrument run logs.		
7.	Inter-element and background correction factors.		
8.	Instrumental data records.		

APPENDIX A-2
LABORATORY REPORT DELIVERABLES CHECKLIST
EXPANDED DATA REPORT

Laboratory Name: _____
Date and Report No.: _____

	Report Item	Complete Yes/No	Comments
J. ICP/MS DATA			
1.	Initial and continuing calibration standard and blank results and summary.		
2.	Low level check standard (CRQL) results and summary.		
3.	Internal standard intensities.		
4.	Tune results and summary.		
5.	Interference check sample results and summary.		
6.	Serial dilutions and post digestion spike results and summary		
7.	Sample preparation records.		
8.	Instrument run logs.		
9.	Inter-element and background correction factors.		
10.	Instrumental data records.		
K. OTHER METALS DATA			
1.	Initial and continuing calibration standard and blank results and summary.		
2.	Dilution tests and post digestion spike results.		
3.	Sample preparation records.		
4.	Instrument run logs.		
5.	Instrumental data records.		
L. INORGANIC DATA			
1.	Initial and continuing calibration standard and blank results summary.		
2.	Calibration verification.		
3.	Sample preparation records.		
4.	Run logs.		
5.	Instrumental data records.		
M. DOCUMENT CONTROL			
1.	Fully executed original CRA Chain of Custody		
2.	Complete internal laboratory custody documentation.		
3.	Copy of Work Order.		

Review Completed By: _____ Date: _____

PROPRIETARY DOCUMENT

CONESTOGA-ROVERS
& ASSOCIATES

E-Mail Date:
E-Mail To:
c.c.:

E-Mail and Hard Copy if Requested

ANALYTICAL DATA ASSESSMENT AND VALIDATION
SUPPLEMENTAL REMEDIAL INVESTIGATION
SITE NAME
SITE LOCATION
SAMPLING DATE

PREPARED BY:
CONESTOGA-ROVERS & ASSOCIATES
2055 Niagara Falls Blvd., Suite #3
Niagara Falls, New York 14304
Telephone: 716-297-6150 Fax: 716-297-2265
Contact:
Date:
www.CRAworld.com

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1.0 INTRODUCTION

The following document details an assessment and validation of analytical results for samples collected in _____. Analyses were provided by _____Laboratories in _____. Sample identifications (ID), times, dates of collection, and parameters analyzed are summarized in Table 1. A summary of the analytical methods is presented in Table 2. Summaries of the analytical results are presented in Tables 3A, 3B, and 3C.

Evaluation of the data was based on information obtained from the finished data sheets, raw data, Chain of Custody forms, calibration data, blank data, duplicate data and recovery data for matrix, blank, and surrogate spikes. The assessment of analytical and in-house data included checks for: data consistency (by observing comparability of duplicate analyses); adherence to accuracy and precision criteria; transmittal errors; and anomalously high and low parameter values.

The quality assurance/quality control (QA/QC) criteria by which these data have been assessed are outlined in the analytical methods referenced in Table 2 and the documents entitled:

i) "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", United States Environmental Protection Agency (USEPA) 540/R-99-008, October 1999;

ii) "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", USEPA 540/R-94-013, February 1994; and

iii) "Quality Assurance Project Plan (QAPP)", October 2005.

Items i) and ii) will hereinafter be referred to as the "Guidelines".

Full deliverables (including raw data) were provided by _____ for the program. The data quality assessment and validation presented in the following subsections were performed based on the sample results and supporting QA/QC provided.

2.0 SAMPLE HOLDING TIMES

The holding time criteria for the analyses are specified in the QAPP.

Sample Chain of Custody documents and analytical reports were used to determine sample holding times. Most samples were prepared and analyzed within the required holding times. Due to laboratory error, six samples requiring polychlorinated biphenyls (PCB) analyses were not extracted within the holding times. The associated sample results were qualified as estimated, based on the potential low bias (see Table 4).

All samples were properly cooled after sampling and upon receipt at the laboratory.

3.0 GAS CHROMATOGRAPH / MASS SPECTROMETER (GC/MS) TUNING AND MASS CALIBRATION - VOLATILES AND SEMI-VOLATILES

Prior to analysis, GC/MS instrumentation is tuned to ensure optimization over the mass range of interest. To evaluate instrument tuning, Methods TO-15/8260B and 8270C require the analysis of specific tuning compounds bromofluorobenzene (BFB) and decafluorotriphenylphosphine (DFTPP), respectively. The resulting spectra must meet the criteria cited in the methods before analysis is initiated. Analysis of the tuning compound must then be repeated every 12 hours throughout sample analysis to ensure the continued optimization of the instrument.

Tuning compounds were analyzed at the required frequency throughout the volatile and semi-volatile analysis periods. All tuning criteria were met, indicating that proper optimization of the instrumentation was achieved.

4.0 INSTRUMENT CALIBRATION

4.1 GC/MS CALIBRATION - VOLATILES AND SEMI-VOLATILES

4.1.1 INITIAL CALIBRATION

In order to quantify compounds of interest in samples, calibration of the GC/MS over a specific concentration range must be performed. Initially, a minimum of a five-point calibration curve containing all compounds of interest is analyzed. In accordance with the SW-846 methods, linearity of the curve and instrument sensitivity are evaluated against the following criteria:

i) all relative response factors (RRFs) must be greater than or equal to 0.05; and

ii) percent relative standard deviation (%RSD) values must not exceed 30 percent.

Method TO-15 initial calibrations were evaluated using the method criteria. All initial calibration data were reviewed and most met the above criteria. Results associated with outlying RSDs were qualified as estimated (see Table 5).

4.1.2 CONTINUING CALIBRATION

To ensure that instrument calibration is acceptable throughout the sample analysis period, continuing calibration standards must be analyzed and compared to the initial calibration curve every 12 hours. In accordance with the SW-846 methods, the following criteria are employed to evaluate continuing calibration data:

i) all RRF values must be greater than or equal to 0.05; and

ii) percent difference (%D) values must not exceed 25 percent.

Method TO-15 continuing calibrations were evaluated using the method criteria. Most continuing calibration results were acceptable. Results associated with outlying %Ds were qualified as estimated (see Table 6).

4.2 GC CALIBRATION - TOTAL CHLORINATED PESTICIDES/PCBs

To ensure that instrument performance was acceptable throughout the pesticide/PCB analysis, the criteria outlined in the methods for initial and continuing instrument calibration have been evaluated.

4.2.1 INSTRUMENT PERFORMANCE

4.2.1.1 PERFORMANCE EVALUATION MIXTURES (PEM)

The PEM is analyzed at the beginning and end of the initial calibration sequence and throughout the analytical sequence. The results of these analyses are used to evaluate dichlorodiphenyltrichloroethane (DDT)/endrin breakdown, using the Method 8081A degradation criteria of $\leq$15 percent. PEM standards were analyzed at the required frequency throughout sample analysis and all method performance criteria were met.

4.2.1.2 INITIAL CALIBRATION

In order to quantify compounds of interest, calibration of the gas chromatograph/electron capture detector (GC/ECD) over a specific concentration range must be performed. Initially, a calibration curve consisting of a minimum of five concentration levels is analyzed for all single component compounds of interest and for Aroclors 1016 and 1260. A single calibration standard is analyzed for all other multi-response compounds. Linearity of the calibration curves is acceptable if all RSD values are less than or equal to 20.0 percent.

Retention time windows are also calculated from the initial calibration analyses. These windows are then used to identify all compounds of interest in subsequent analyses.

All initial calibration standards were analyzed at the required frequencies. All retention time, peak resolution and linearity criteria were satisfied as specified in the methods.

4.2.1.3 CONTINUING CALIBRATION

To ensure that the calibration of the instrument is valid throughout the sample analysis period, continuing calibration standards are analyzed and evaluated on a regular basis.

To evaluate the continued linearity of the calibration, %D values are calculated for each compound. As specified in the methods, all %D values should be less than 15 percent.

To ensure that compound retention times do not vary over the analysis period, all retention times for continuing calibration compounds must fall within the established retention time windows.

All continuing calibration standards were analyzed at the required frequency. Most %D values and all compound retention times met the above criteria indicating acceptable instrument calibration throughout the analysis period. Results associated with outlying %Ds were qualified as estimated (see Table 6).

4.3 INORGANICS CALIBRATION

4.3.1 INSTRUMENT CALIBRATION

4.3.1.1 INITIAL CALIBRATION

Initial calibration of the instruments ensures that they are capable of producing satisfactory quantitative data at the beginning of a series of analyses. For Inductively Coupled Plasma (ICP) analysis, a calibration blank and at least one standard must be analyzed at each wavelength to establish the analytical curve. For mercury Atomic Absorption (AA) analyses, a calibration blank and a minimum of five standards must be analyzed to establish the analytical curve and resulting correlation coefficients must be at least 0.995. For cyanide analyses, a calibration blank and a minimum of four standards must be analyzed to establish the analytical curve and resulting correlation coefficients must be at least 0.995.

After the analyses of the calibration curves, an Initial Calibration Verification (ICV) standard must be analyzed to verify the analytical accuracy of the calibration curves. All analyte recoveries from the analyses of the ICVs must be within the following control limits.

Analytical Method	*Parameter*	*Control Limits*
ICP/AA	Metals	90 - 110%
Cold Vapor AA	Mercury	80 - 120%
Instrumental Wet Chemistry	Cyanide	85 - 115%

Upon review of the data, it was determined that the calibration curves and ICVs were analyzed at the proper frequencies and that all of the above-specified criteria were met. The laboratory effectively demonstrated that the instrumentation used for metals and cyanide analyses was properly calibrated prior to sample analyses.

4.3.1.2 CONTINUING CALIBRATION

To ensure that instrument calibration is acceptable throughout the sample analysis period, Continuing Calibration Verification (CCV) standards are analyzed on a regular basis. Each CCV is deemed acceptable if all analyte recoveries are within the control limits specified above for the ICVs. If some of the CCV analyte recoveries are outside the control limits, samples analyzed before and after the CCV, up until the previous and proceeding CCV analyses, are affected.

For this study, CCVs were analyzed at the proper frequency. All analyte recoveries reported for the CCVs were within the specified limits.

5.0 INTERNAL STANDARDS ANALYSES

Internal standard data were evaluated for all total volatile and semi-volatile samples.

5.1 VOLATILES

To ensure that changes in the GC/MS sensitivity and response do not affect sample analysis results, internal standard compounds are added to each sample prior to analysis. All results are then calculated as a ratio of the internal standard responses.

The sample internal standard results for Method 8260B were evaluated against the following criteria:

i) the retention time of the internal standard must not vary more than ±30 seconds from the associated calibration standard; and

ii) internal standard area counts must not vary by more than a factor of two (-50 percent to +100 percent) from the associated calibration standard.

Method TO-15 internal standards were evaluated using the method criteria. Most internal standard recoveries associated with the reported sample results and retention times reported met the above criteria. Results associated with outlying area counts were qualified as estimated (see Table 7).

5.2 SEMI-VOLATILES

To ensure that changes in the GC/MS sensitivity and response do not affect sample analysis results, internal standard compounds are added to each sample prior to analysis. All results are then calculated as a ratio of the internal standard responses.

The sample internal standard results were evaluated against the following criteria:

i) the retention time of the internal standard must not vary more than ±30 seconds from the associated calibration standard; and

ii) internal standard area counts must not vary by more than a factor of two (-50 percent to +100 percent) from the associated calibration standard.

Most associated internal standard recoveries and retention times reported met the above criteria. A poor chrysene-d12 response (<10 percent) was reported, and the associated non-detect sample results were rejected. Remaining results associated with outlying area counts were qualified as estimated (see Table 7).

6.0 SURROGATE SPIKE RECOVERIES

In accordance with the methods employed, all samples, blanks and standards analyzed for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), chlorinated pesticides, and PCBs were spiked with surrogate compounds prior to sample extraction and/or analysis. Surrogate recoveries provide a means to evaluate the effects of individual sample matrices on analytical efficiency. Non-detect sample results associated with high surrogate recoveries were not qualified, as the indicated high bias would not impact the data. All other surrogate recoveries were evaluated as specified below.

6.1 VOLATILES

All samples submitted for VOC determinations were spiked with surrogate compounds prior to sample analysis. All surrogate recoveries were within the laboratory control limits.

6.2 SEMI-VOLATILES

All samples submitted for analysis were spiked with surrogate compounds prior to sample extraction and analysis. According to the "Guidelines", up to one outlying surrogate in the base/neutral or acid fractions is acceptable as long as the recovery is at least 10 percent.

All sample analyses yielded acceptable recoveries.

6.3 CHLORINATED PESTICIDES/PCBs

All investigative samples submitted for chlorinated pesticide/PCB determinations were spiked with the surrogate compounds tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) prior to sample preparation. Surrogate recoveries could not be assessed for some analyses due to necessary sample dilutions. All remaining results associated with outlying DCB and TCMX surrogate recoveries were qualified as estimated (see Table 8).

7.0 LABORATORY BLANK ANALYSES

The purpose of assessing the results of laboratory blank analyses is to determine the existence and magnitude of sample contamination introduced during analysis. Laboratory blanks are prepared and analyzed as samples.

For this study, laboratory blanks were analyzed at a minimum frequency of one per 20 investigative samples and/or one per analytical batch.

7.1 VOLATILES

Most volatile analytes of interest were reported as non-detect in the method blanks. Low VOC concentrations were reported for some analyses. All associated samples with similar concentrations were qualified as non-detect (see Table 9).

7.2 SEMI-VOLATILES

Most semi-volatile analytes of interest were reported as non-detect in the method blanks. Low SVOC concentrations were reported for some analyses. All associated samples with similar concentrations were qualified as non-detect (see Table 9).

7.3 CHLORINATED PESTICIDES/PCBs

Analysis of the laboratory blanks yielded non-detect results for all pesticides and PCBs indicating contamination was not a factor for this analysis.

7.4 INORGANIC ANALYSES

Upon review of the initial calibration blanks, continuing calibration blanks and preparation blanks, it was noted that metal concentrations were observed above the instrument detection limit (IDL). Most investigative samples associated with contaminated laboratory blanks yielded either non-detect concentrations or concentrations greater than five times the associated laboratory blank concentrations for the analytes of interest. These sample results were not impacted by the contamination detected. Associated sample results that were impacted were qualified as non-detect (see Table 9).

8.0 LABORATORY CONTROL SAMPLE (LCS) ANALYSES

LCS and/or laboratory control sample duplicates (LCSD) are prepared and analyzed as samples to assess the analytical efficiencies of the methods employed, independent of sample matrix effects.

LCS analyses were performed for all parameters at a minimum frequency of one per 20 investigative samples.

8.1 VOLATILES

Blank samples were spiked with the specified compounds. Most blank spike sample analyses yielded recoveries within the laboratory control limits. Low 1,2,4-trichlorobenzene recoveries were reported for the air analyses, and the associated sample results were qualified as estimated (see Table 10).

APPENDIX B-1

FULL DATA VALIDATION REPORT TEMPLATE

8.2 SEMI-VOLATILES

Blank samples were spiked with the specified SVOC compounds prior to extraction. All recoveries reported for the blank spikes were within the control limits, indicating that acceptable laboratory accuracy was achieved during the analysis.

8.3 CHLORINATED PESTICIDES/PCBs

LCS samples were spiked with the specified compounds. All recoveries reported for the blank spikes were within the control limits, indicating acceptable analytical accuracy.

8.4 INORGANICS

The LCS serves as a monitor of the overall performance of all steps in the analysis, indicating the sample preparation. LCSs were analyzed using the same sample preparation, analytical methods, and QA/QC procedures employed for the inorganic samples.

All LCS samples yielded recoveries within the established control limits, indicating acceptable laboratory performance.

9.0 MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD) ANALYSES

To evaluate the effects of sample matrices on the extraction or digestion process, measurement procedures, and accuracy of a particular analysis, samples are spiked with a known concentration of the analyte of concern and analyzed as MS/MSD samples. The relative percent difference (RPD) between the MS and MSD are used to assess analytical precision. Non-detect sample results associated with high MS/MSD recoveries were not qualified, as the indicated high bias would not impact the data. MS/MSD analyses were performed as specified in Table 1.

9.1 VOLATILES

The MS/MSD samples were spiked with representative compounds. Most percent recoveries and RPD values were within the laboratory control limits. Results associated with outlying MS/MSD recoveries were qualified as estimated (see Table 11).

9.2 SEMI-VOLATILES

The MS/MSD samples were spiked with representative compounds prior to extraction. Most percent recoveries and RPD values were within the laboratory control limits. Results associated with outlying MS/MSD recoveries were qualified as estimated (see Table 11).

9.3 CHLORINATED PESTICIDES/PCBs

The MS/MSD samples were spiked with representative chlorinated pesticide compounds. No qualification of sample data was necessary for this analysis.

9.4 INORGANICS

The MS/MSD samples were spiked with cyanide and the metals of interest. Per the "Guidelines", the data cannot be assessed if the sample concentration exceeds four times the spike concentration.

Most MS/MSD recoveries were acceptable. Results associated with outlying MS/MSD recoveries were qualified as estimated (see Table 11).

10.0 ICP SERIAL DILUTION

The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix. A minimum of one per 20 investigative samples or at least one per analytical batch must be analyzed at a five-fold dilution. For samples yielding analyte concentrations greater than 50 times the IDL, the serial dilution results must agree within 10 percent of the original results.

A serial dilution was performed on the MS sample. Most results met the criteria above. Results associated with outlying analyses were qualified as estimated (see Table 12).

11.0 ICP INTERFERENCE CHECK SAMPLE ANALYSIS (ICS)

To verify that the laboratory has established proper interelement and background correction factors, ICSs are analyzed. These samples contain high concentrations of aluminum, calcium, magnesium and iron and are analyzed at the beginning and end of each sample analysis period.

ICS analysis results were evaluated for all samples. All ICS recoveries were within the established control limits of 80-120 percent.

12.0 FIELD QA/QC

12.1 FIELD DUPLICATES

To assess the analytical and sampling protocol precision, five field duplicates (as identified in Table 1) was collected and submitted "blind" to the laboratory. The results were evaluated using the criteria in the QAPP. Most field duplicate results showed adequate reproducibility outside of the estimated region of detection. Results that did show variability were qualified as estimated (see Table 13).

12.2 RINSE BLANKS

To assess contamination from field equipment cleaning activities, two rinse blanks were collected as identified in Table 1. Most sample results were non-detect for the analytes of interest. Results impacted by rinse blank contamination were qualified as non-detect (see Table 14).

12.3 TRIP BLANKS

Trip blanks are transported, stored, and analyzed with the investigative samples to identify potential cross-contamination of VOCs. Three trip blanks were collected as identified in Table 1. Most sample results were non-detect for the analytes of interest. Results impacted by trip blank contamination were qualified as non-detect (see Table 15).

13.0 SAMPLE QUANTITATION

All soil results were reported on a dry weight basis.

The laboratory confirmed all detected pesticide/PCB results using a second column. In some instances, there was notable variability between the results from each column. In these cases, the higher of the two results was reported, and the associated sample results were qualified as estimated (see Table 16).

14.0 TENTATIVELY IDENTIFIED COMPOUNDS (TICs)

Chromatographic peaks recorded during volatile and semi-volatile sample analyses that are not target compounds, surrogates, or internal standards, are potential TICs.

Summaries of the TICs reported by the laboratory are presented in Tables 17A and 17B.

15.0 CONCLUSION

Based on this QA/QC assessment, the data produced by ____ are acceptable with the specific exceptions and qualifications noted.

PROPRIETARY DOCUMENT

TABLES

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TABLE 1

SAMPLE COLLECTION AND ANALYSIS SUMMARY
SUPPLEMENTAL REMEDIAL INVESTIGATION

					Analysis/Parameters					
Sample ID	*Location ID*	*Matrix*	*Collection Date (mm/dd/yy)*	*Collection Time (hr:min)*	*TCL VOCs + TICs*	*TCL SVOCs + TICs and Dioxin Screen*	*TCL Pesticides/PCBs*	*TAL Metals/Cyanide*	*TO-15 VOCs*	*Comments*
W-42271-102505-WA-01	SV-18	Soil/waste	10/25/05	16:30	X	X	X	X	X	
S-42271-102605-WA-02	SV-17	Soil/surface	10/26/05	10:15	X	X	X	X	X	
S-42271-102605-WA-03	SV-17	Soil/fill	10/26/05	10:25	X	X	X	X	X	
W-42271-102605-WA-04	SV-17	Soil/waste	10/26/05	10:35	X	X	X	X	X	
S-42271-102605-WA-06	MW-3-05	Soil/surface	10/26/05	13:05	X	X	X	X	X	
W-42271-102605-WA-07	MW-3-05	Soil/waste	10/26/05	13:23	X	X	X	X	X	
S-42271-102605-WA-08	MW-5-05	Soil/surface	10/26/05	14:35	X	X	X	X	X	
S-42271-102705-WA-09	SV-16	Soil/surface	10/27/05	8:45	X	X	X	X	X	
S-42271-102705-WA-09-D	SV-16	Soil/surface	10/27/05	8:50	X	X	X	X	X	Field Duplicate of S-42271-102705-WA-09
W-42271-102705-WA-10	SV-16	Soil/waste	10/27/05	9:20	X	X	X	X	X	
W-42271-102705-WA-10-D	SV-16	Soil/waste	10/27/05	9:20	X	X	X	X	X	Field Duplicate of W-42271-102705-WA-10

Notes:
- Not applicable.
MS Matrix Spike.
MSD Matrix Spike Duplicate.
SVOCs Semi-Volatile Organic Compounds.
TAL Target Analyte List.
TCL Target Compound List.
TICs Tentatively Identified Compounds.
VOCs Volatile Organic Compounds.

PROPRIETARY DOCUMENT

TABLE 2

ANALYTICAL METHODS SUMMARY
SUPPLEMENTAL REMEDIAL INVESTIGATION

Analytical Parameters	*Analytical Method*
TCL VOCs (Plus TICs)	8260 [1]
TCL SVOCs (with 1,4-Dioxane, TICs, and Dioxin Screen)	8270 [1]
TCL Pesticides/PCBs	8081/8082 [1]
TAL Inorganics (Soil)	6010/7471/9012 [1]
TAL Inorganics (Water)	6010/7470/9012 [1]
Percent Solids	160.3 [2]
VOCs	TO-15 [3]

Notes:

(1) "Test Methods for Evaluating Solid Waste", SW-846, 3rd Edition (with revisions).

(2) "Methods for the Chemical Analysis of Water and Wastes", USEPA 600/4-79-220, March 1983 (with revisions).

(3) "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air", January 1999.

PCBs Polychlorinated Biphenyls.

SVOCs Semi-Volatile Organic Compounds.

TAL Target Analyte List.

TCL Target Compound List.

TICs Tentatively Identified Compounds.

VOCs Volatile Organic Compounds.

TABLE 3A

ANALYTICAL RESULTS SUMMARY - SOIL SAMPLING
SUPPLEMENTAL REMEDIAL INVESTIGATION

	Sample Location:	*MW-3-05*	*MW-3-05*	*MW-5-05*	*MW-7-05*	*MW-9-05*	*POND*	*Low lying area near RCR Yacht*
	Sample ID:	*S-42271-102605-WA-06*	*S-42271-102605-WA-07*	*S-42271-102605-WA-08*	*S-42271-110105-WA-21*	*S-42271-110105-WA-18*	*S-42271-110305-WA-027*	*S-42271-110305-WA-025*
	Sample Date	*10/26/2005*	*10/26/2005*	*10/26/2005*	*11/1/2005*	*11/1/2005*	*11/3/2005*	*11/3/2005*
Parameter	*Units*							
Volatile Organic Compounds								
1,1,1-Trichloroethane	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
1,1,2,2-Tetrachloroethane	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
1,1,2-Trichloroethane	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
1,1-Dichloroethane	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
1,1-Dichloroethene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
1,2,4-Trichlorobenzene	µg/kg	5.3 U	320 U	5.8 UJ	5.6 U	5.5 U	7.5 UJ	7.5 UJ
1,2-Dibromo-3-chloropropane (DBCP)	µg/kg	11 UJ	640 U	12 UJ	11 UJ	11 UJ	15 UJ	15 UJ
1,2-Dibromoethane (Ethylene Dibromide)	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
1,2-Dichlorobenzene	µg/kg	5.3 U	24 J	5.8 UJ	5.6 U	5.5 U	7.5 UJ	7.5 UJ
1,2-Dichloroethane	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
1,2-Dichloropropane	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
1,3-Dichlorobenzene	µg/kg	5.3 U	320 U	5.8 UJ	5.6 U	5.5 U	7.5 UJ	7.5 UJ
1,4-Dichlorobenzene	µg/kg	5.3 U	320 U	5.8 UJ	5.6 U	5.5 U	7.5 UJ	7.5 UJ
2-Butanone (Methyl Ethyl Ketone)	µg/kg	21 U	1300 U	23 U	22 U	9.5 J	23 J	3.9 J
2-Hexanone	µg/kg	21 U	1300 U	23 U	22 U	22 U	30 UJ	30 UJ
4-Methyl-2-Pentanone (Methyl Isobutyl Ketone)	µg/kg	21 U	1300 U	23 U	22 U	22 U	30 U	30 U
Acetone	µg/kg	21 U	1300 U	23 U	22 U	22 U	21 J	15 J
Benzene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	0.75 J
Bromodichloromethane	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Bromoform	µg/kg	5.3 UJ	320 U	5.8 UJ	5.6 UJ	5.5 UJ	7.5 U	7.5 U
Bromomethane (Methyl Bromide)	µg/kg	5.3 UJ	320 U	5.8 UJ	5.6 UJ	5.5 UJ	7.5 U	7.5 U
Carbon disulfide	µg/kg	5.3 U	25 J	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Carbon tetrachloride	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Chlorobenzene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Chloroethane	µg/kg	5.3 UJ	320 U	5.8 UJ	5.6 UJ	5.5 UJ	7.5 U	7.5 U
Chloroform (Trichloromethane)	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Chloromethane (Methyl Chloride)	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
cis-1,2-Dichloroethene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
cis-1,3-Dichloropropene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Cyclohexane	µg/kg	11 U	640 U	12 U	11 U	11 U	15 U	15 U
Dibromochloromethane	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Dichlorodifluoromethane (CFC-12)	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Ethylbenzene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Isopropylbenzene	µg/kg	0.67 J	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Methyl acetate	µg/kg	11 U	640 U	12 U	11 U	11 U	15 U	15 U
Methyl cyclohexane	µg/kg	11 U	640 U	12 U	11 U	11 U	15 U	15 U
Methyl Tert Butyl Ether	µg/kg	21 U	1300 U	23 U	22 U	22 U	30 U	30 U
Methylene chloride	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Styrene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Tetrachloroethene	µg/kg	0.98 J	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Toluene	µg/kg	5.3 U	20 J	5.8 U	5.6 U	5.5 U	6.5 J	0.44 J
trans-1,2-Dichloroethene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
trans-1,3-Dichloropropene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Trichloroethene	µg/kg	5.3 U	320 U	5.8 U	5.6 U	5.5 U	7.5 U	7.5 U
Trichlorofluoromethane (CFC-11)	µg/kg	5.3 UJ	320 U	5.8 UJ	5.6 U	5.5 U	7.5 U	7.5 U

TABLE 3A

ANALYTICAL RESULTS SUMMARY - SOIL SAMPLING
SUPPLEMENTAL REMEDIAL INVESTIGATION

	Sample Location:	*MW-3-05*	*MW-3-05*	*MW-5-05*	*MW-7-05*	*MW-9-05*	*POND*	*Low lying area near RCR Yacht*
	Sample ID:	*S-42271-102605-WA-06*	*S-42271-102605-WA-07*	*S-42271-102605-WA-08*	*S-42271-110105-WA-21*	*S-42271-110105-WA-18*	*S-42271-110305-WA-027*	*S-42271-110305-WA-025*
	Sample Date	*10/26/2005*	*10/26/2005*	*10/26/2005*	*11/1/2005*	*11/1/2005*	*11/3/2005*	*11/3/2005*
Parameter	*Units*							
Wet Chemistry								
Cyanide (total)	mg/kg	2.1	0.44 J	0.23 J	1.2	0.61	7.6	11.5 J
Total Solids	%	80.1	78.7	84.8	82.5	91.6	64.4	61.2

Notes:

J Estimated.

U Non-detect at associated value.

UJ The analyte was not detected above the sample quantitation limit. The reported quantitation limit is an estimated quantity.

PROPRIETARY DOCUMENT

TABLE 4

QUALIFIED SAMPLE RESULTS DUE TO HOLDING TIME EXCEEDANCES
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Sample ID*	*Holding Time (days)*	*Holding Time Criteria (days)*	*Analyte*	*Qualified Sample Results*	*Units*
PCBs	S-42271-102705-WA-12	21	10	Aroclor-1232 (PCB-1232)	48 UJ	µg/Kg
				Aroclor-1248 (PCB-1248)	48 UJ	µg/Kg
				Aroclor-1016 (PCB-1016)	48 UJ	µg/Kg
				Aroclor-1242 (PCB-1242)	48 UJ	µg/Kg
				Aroclor-1260 (PCB-1260)	48 UJ	µg/Kg
				Aroclor-1254 (PCB-1254)	17 J	µg/Kg
				Aroclor-1221 (PCB-1221)	48 UJ	µg/Kg
PCBs	S-42271-102705-WA-13	21	10	Aroclor-1260 (PCB-1260)	39 UJ	µg/Kg
				Aroclor-1254 (PCB-1254)	39 UJ	µg/Kg
				Aroclor-1221 (PCB-1221)	39 UJ	µg/Kg
				Aroclor-1232 (PCB-1232)	39 UJ	µg/Kg
				Aroclor-1248 (PCB-1248)	39 UJ	µg/Kg
				Aroclor-1016 (PCB-1016)	39 UJ	µg/Kg
				Aroclor-1242 (PCB-1242)	39 UJ	µg/Kg
PCBs	S-42271-102805-WA-14	21	10	Aroclor-1260 (PCB-1260)	53 UJ	µg/Kg
				Aroclor-1254 (PCB-1254)	53 UJ	µg/Kg
				Aroclor-1221 (PCB-1221)	53 UJ	µg/Kg
				Aroclor-1232 (PCB-1232)	53 UJ	µg/Kg
				Aroclor-1248 (PCB-1248)	53 UJ	µg/Kg
				Aroclor-1016 (PCB-1016)	53 UJ	µg/Kg
				Aroclor-1242 (PCB-1242)	53 UJ	µg/Kg
PCBs	S-42271-103105-WA-15	21	10	Aroclor-1260 (PCB-1260)	37 UJ	µg/Kg
				Aroclor-1254 (PCB-1254)	37 UJ	µg/Kg
				Aroclor-1221 (PCB-1221)	37 UJ	µg/Kg
				Aroclor-1232 (PCB-1232)	37 UJ	µg/Kg
				Aroclor-1248 (PCB-1248)	37 UJ	µg/Kg
				Aroclor-1016 (PCB-1016)	37 UJ	µg/Kg
				Aroclor-1242 (PCB-1242)	37 UJ	µg/Kg
PCBs	S-42271-103105-WA-16	21	10	Aroclor-1260 (PCB-1260)	41 UJ	µg/Kg
				Aroclor-1254 (PCB-1254)	41 UJ	µg/Kg
				Aroclor-1221 (PCB-1221)	41 UJ	µg/Kg
				Aroclor-1232 (PCB-1232)	41 UJ	µg/Kg
				Aroclor-1248 (PCB-1248)	41 UJ	µg/Kg
				Aroclor-1016 (PCB-1016)	41 UJ	µg/Kg
				Aroclor-1242 (PCB-1242)	41 UJ	µg/Kg
PCBs	S-42271-103105-WA-17	21	10	Aroclor-1260 (PCB-1260)	40 UJ	µg/Kg
				Aroclor-1254 (PCB-1254)	40 UJ	µg/Kg
				Aroclor-1221 (PCB-1221)	40 UJ	µg/Kg
				Aroclor-1232 (PCB-1232)	40 UJ	µg/Kg
				Aroclor-1248 (PCB-1248)	40 UJ	µg/Kg
				Aroclor-1016 (PCB-1016)	40 UJ	µg/Kg
				Aroclor-1242 (PCB-1242)	40 UJ	µg/Kg

Notes:
J Estimated.
PCBs Polychlorinated Biphenyls.
UJ The analyte was not detected above the sample quantitation limit. The reported quantitation limit is an estimated quantity.

PROPRIETARY DOCUMENT

TABLE 5

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING INITIAL CALIBRATION RESULTS
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Compound*	*Calibration Dates*	*%RSD*	*Associated Sample ID*	*Qualified Sample Results*	*Units*
VOCs in Air	2-Butanone (Methyl Ethyl Ketone)	11/03/05	33	A-42271-JC-001	0.88 J	ppbv
				A-42271-JC-002	1.0 J	ppbv
				A-42271-JC-003	2.8 J	ppbv
				A-42271-JC-004	2.5 UJ	ppbv
				A-42271-JC-005	16 J	ppbv
				A-42271-JC-006	0.82 J	ppbv
				A-42271-JC-007	0.50 UJ	ppbv
				A-42271-JC-008	5.0 UJ	ppbv
				A-42271-JC-009	5.0 UJ	ppbv
				A-42271-JC-010	0.50 UJ	ppbv
				A-42271-JC-011	0.50 UJ	ppbv
				A-42271-JC-012	5.0 UJ	ppbv
				A-42271-JC-013	3.0 J	ppbv
				A-42271-JC-014	0.50 UJ	ppbv
				A-42271-JC-015	1.5 UJ	ppbv
				A-42271-JC-016	21 UJ	ppbv
				A-42271-JC-017	5.0 UJ	ppbv
				A-42271-JC-018	3.6 J	ppbv
				A-42271-JC-019	0.50 UJ	ppbv
				A-42271-JC-020	0.50 UJ	ppbv

Notes:
%RSD Percent Relative Standard Deviation.
J Estimated.
UJ The analyte was not detected above the sample quantitation limit. The reported quantitation limit is an estimated quantity.
VOCs Volatile Organic Compounds.

PROPRIETARY DOCUMENT

TABLE 6

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING CONTINUING CALIBRATION RESULTS
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Calibration Date*	*Compound*	*%D*	*Associated Sample ID*	*Qualified Sample Results*	*Units*
VOCs in Air	11/12/05	Acetone	31	A-42271-JC-016	210 UJ	ppbv
Volatiles	11/07/05	Bromomethane (Methyl Bromide)	-61	W-42271-102505-WA-01	6.0 UJ	µg/Kg
				S-42271-102605-WA-02	5.9 UJ	µg/Kg
				S-42271-102605-WA-03	7.1 UJ	µg/Kg
				S-42271-102605-WA-06	5.3 UJ	µg/Kg
				S-42271-102605-WA-08	5.8 UJ	µg/Kg
Volatiles	11/07/05	Chloroethane	-43	W-42271-102505-WA-01	6.0 UJ	µg/Kg
				S-42271-102605-WA-02	5.9 UJ	µg/Kg
				S-42271-102605-WA-03	7.1 UJ	µg/Kg
				S-42271-102605-WA-06	5.3 UJ	µg/Kg
				S-42271-102605-WA-08	5.8 UJ	µg/Kg
Volatiles	11/07/05	Trichlorofluoromethane (CFC-11)	-29	W-42271-102505-WA-01	6.0 UJ	µg/Kg
				S-42271-102605-WA-02	5.9 UJ	µg/Kg
				S-42271-102605-WA-03	7.1 UJ	µg/Kg
				S-42271-102605-WA-06	5.3 UJ	µg/Kg
				S-42271-102605-WA-08	5.8 UJ	µg/Kg
Volatiles	11/07/05	Bromoform	31	W-42271-102505-WA-01	6.0 UJ	µg/Kg
				S-42271-102605-WA-02	5.9 UJ	µg/Kg
				S-42271-102605-WA-03	7.1 UJ	µg/Kg
				S-42271-102605-WA-06	5.3 UJ	µg/Kg
				S-42271-102605-WA-08	5.8 UJ	µg/Kg
Volatiles	11/07/05	1,2-Dibromo-3-chloropropane (DBCP)	36	W-42271-102505-WA-01	12 UJ	µg/Kg
				S-42271-102605-WA-02	12 UJ	µg/Kg
				S-42271-102605-WA-03	14 UJ	µg/Kg
				S-42271-102605-WA-06	11 UJ	µg/Kg
				S-42271-102605-WA-08	12 UJ	µg/Kg
Volatiles	11/08/05	Bromomethane (Methyl Bromide)	-51	S-42271-102705-WA-09-D	6.9 UJ	µg/Kg
				S-42271-102605-WA-04	6.6 UJ	µg/Kg
Volatiles	11/08/05	Chloroethane	-42	S-42271-102705-WA-09-D	6.9 UJ	µg/Kg
				S-42271-102605-WA-04	6.6 UJ	µg/Kg
Volatiles	11/08/05	Trichlorofluoromethane (CFC-11)	-27	S-42271-102705-WA-09-D	6.9 UJ	µg/Kg
				S-42271-102605-WA-04	6.6 UJ	µg/Kg
Volatiles	11/09/05	Bromomethane (Methyl Bromide)	-36	S-042271-102705-WA-09	6.5 UJ	µg/Kg
				S-42271-102705-WA-10	6.2 UJ	µg/Kg
				S-42271-102705-WA-12	7.1 UJ	µg/Kg
				S-42271-103105-WA-15	5.4 UJ	µg/Kg

Notes:
%D Percent Difference.
UJ The analyte was not detected above the sample quantitation limit. The reported quantitation limit is an estimated quantity.
VOCs Volatile Organic Compounds.

PROPRIETARY DOCUMENT

TABLE 7

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING INTERNAL STANDARD (IS) RECOVERIES
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Sample ID*	*IS*	*IS Area Count (percent)*	*Control Limits (percent)*	*Analytes*	*Qualified Sample Results*	*Units*
Volatiles	S-42271-110305-WA-025	1,4-Dichlorobenzene-d4	42	50-200	1,4-Dichlorobenzene	7.5 UJ	µg/Kg
					1,2,4-Trichlorobenzene	7.5 UJ	µg/Kg
					1,3-Dichlorobenzene	7.5 UJ	µg/Kg
					1,2-Dichlorobenzene	7.5 UJ	µg/Kg
					1,2-Dibromo-3-chloropropane (DBCP)	15 UJ	µg/Kg
Volatiles	S-42271-110305-WA-026	1,4-Dichlorobenzene-d4	39	50-200	1,4-Dichlorobenzene	8.1 UJ	µg/Kg
					1,2,4-Trichlorobenzene	8.1 UJ	µg/Kg
					1,3-Dichlorobenzene	8.1 UJ	µg/Kg
					1,2-Dichlorobenzene	8.1 UJ	µg/Kg
					1,2-Dibromo-3-chloropropane (DBCP)	16 UJ	µg/Kg
Volatiles	S-42271-110305-WA-027	1,4-Dichlorobenzene-d4	48	50-200	1,4-Dichlorobenzene	7.5 UJ	µg/Kg
					1,2,4-Trichlorobenzene	7.5 UJ	µg/Kg
					1,3-Dichlorobenzene	7.5 UJ	µg/Kg
					1,2-Dichlorobenzene	7.5 UJ	µg/Kg
					1,2-Dibromo-3-chloropropane (DBCP)	15 UJ	µg/Kg
Volatiles	S-42271-110105-WA-19	1,4-Dichlorobenzene-d4	42	50-200	1,4-Dichlorobenzene	5.5 UJ	µg/Kg
					1,2,4-Trichlorobenzene	5.5 UJ	µg/Kg
					1,3-Dichlorobenzene	5.5 UJ	µg/Kg
					1,2-Dichlorobenzene	5.5 UJ	µg/Kg
					1,2-Dibromo-3-chloropropane (DBCP)	11 UJ	µg/Kg
Volatiles	W-42271-110205-WA-24	1,4-Dichlorobenzene-d4	36	50-200	1,4-Dichlorobenzene	6.8 UJ	µg/Kg
					1,2,4-Trichlorobenzene	6.8 UJ	µg/Kg
					1,3-Dichlorobenzene	6.8 UJ	µg/Kg
					1,2-Dichlorobenzene	6.8 UJ	µg/Kg
					1,2-Dibromo-3-chloropropane (DBCP)	14 UJ	µg/Kg

Notes:
UJ The analyte was not detected above the sample quantitation limit. The reported quantitation limit is an estimated quantity.

TABLE 8

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING SURROGATE RECOVERIES
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Surrogate*	*Surrogate Recovery (percent)*	*Control Limits (percent)*	*Sample ID*	*Analytes*	*Qualified Sample Results*	*Units*
PCBs	DCB	18	40 - 138	S-42271-102605-WA-02	Aroclor-1260 (PCB-1260)	14 J	µg/Kg
					Aroclor-1260 (PCB-1260)	42 UJ	µg/Kg
					Aroclor-1254 (PCB-1254)	42 UJ	µg/Kg
					Aroclor-1221 (PCB-1221)	42 UJ	µg/Kg
					Aroclor-1232 (PCB-1232)	37 J	µg/Kg
					Aroclor-1248 (PCB-1248)	42 UJ	µg/Kg
					Aroclor-1016 (PCB-1016)	42 UJ	µg/Kg
					Aroclor-1242 (PCB-1242)		
PCBs	DCB	175	40 - 138	S-42271-102605-WA-04	Aroclor-1260 (PCB-1260)	370 J	µg/Kg
					Aroclor-1248 (PCB-1248)	590 J	µg/Kg
PCBs	DCB	167	40 - 138	S-42271-102605-WA-09-D	Aroclor-1260 (PCB-1260)	55 J	µg/Kg
					Aroclor-1248 (PCB-1248)	120 J	µg/Kg
Pesticides	TCMX	37	39 - 130	W-42271-110405-WA-05	Heptachlor epoxide	0.050 UJ	µg/L
					Endosulfan sulfate	0.050 UJ	µg/L
					Aldrin	0.050 UJ	µg/L
					alpha-BHC	0.050 UJ	µg/L
					beta-BHC	0.050 UJ	µg/L
					delta-BHC	0.050 UJ	µg/L
					Endosulfan II	0.050 UJ	µg/L
					4,4'-DDT	0.050 UJ	µg/L

Notes:
DCB Decachlorobiphenyl.
J Estimated.
PCBs Polychlorinated Biphenyls.
TCMX Tetrachloro-m-xylene.
UJ The analyte was not detected above the sample quantitation limit. The reported quantitation limit is an estimated quantity.

PROPRIETARY DOCUMENT

TABLE 9

QUALIFIED SAMPLE RESULTS DUE TO ANALYTE CONCENTRATIONS IN THE LABORATORY BLANKS
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Analysis Date*	*Analyte*	*Blank Result* [1]	*Sample ID*	*Qualified Sample Result*	*Units*
Volatiles	11/07/05	1,2-Dichlorobenzene	0.30J	S-42271-102605-WA-06	5.3 U	µg/Kg
Volatiles	11/07/05	1,4-Dichlorobenzene	0.46J	S-42271-102605-WA-06	5.3 U	µg/Kg
Volatiles	11/08/05	Methylene chloride	3.0J	S-42271-102605-WA-04	6.6 U	µg/Kg
Volatiles	11/10/05	Acetone	110J	S-42271-102605-WA-07	1300 U	µg/Kg
Volatiles	11/10/05	1,4-Dichlorobenzene	12J	S-42271-102605-WA-07	320 U	µg/Kg
Volatiles	11/10/05	Methyl acetate	67J	S-42271-102705-WA-10-D	620 U	µg/Kg
				S-42271-102605-WA-07	640 U	µg/Kg
				S-42271-102705-WA-11	590 U	µg/Kg
Volatiles	11/10/05	Methylene chloride	170J	S-42271-102705-WA-10-D	310 U	µg/Kg
				S-42271-102605-WA-07	320 U	µg/Kg
				S-42271-102705-WA-11	300 U	µg/Kg
Volatiles	11/10/05	1,2,4-Trichlorobenzene	24J	S-42271-102705-WA-10-D	310 U	µg/Kg
				S-42271-102605-WA-07	320 U	µg/Kg
Volatiles	11/09/05	1,2-Dichlorobenzene	0.28J	S-42271-102705-WA-12	7.1 U	µg/Kg
Volatiles	11/09/05	1,4-Dichlorobenzene	0.40J	S-42271-102705-WA-10	6.2 U	µg/Kg
Volatiles	11/09/05	1,2,4-Trichlorobenzene	0.79J	S-42271-102705-WA-12	7.1 U	µg/Kg
Volatiles	11/10/05	Methylene chloride	1.9J	S-42271-103105-WA-17	5.9 U	µg/Kg
Volatiles	11/10/05	Carbon disulfide	0.31J	S-42271-110305-WA-025	7.5 U	µg/Kg
				S-42271-110305-WA-027	7.5 U	µg/Kg
				W-42271-110205-WA-22	5.1 U	µg/Kg
				S-42271-110305-WA-026	8.1 U	µg/Kg
Volatiles	11/10/05	Methylene chloride	3.3J	S-42271-110305-WA-025	7.5 U	µg/Kg
				S-42271-110305-WA-027	7.5 U	µg/Kg
				S-42271-110305-WA-026	8.1 U	µg/Kg
Volatiles	11/11/05	Carbon disulfide	0.27J	S-42271-110105-WA-19	5.5 U	µg/Kg
				S-42271-110205-WA-23	5.9 U	µg/Kg
				W-42271-110105-WA-20	6.1 U	µg/Kg
				W-42271-110205-WA-24	6.8 U	µg/Kg
Volatiles	11/11/05	Methylene chloride	4.4J	S-42271-110105-WA-19	5.5 U	µg/Kg
				S-42271-110205-WA-23	5.9 U	µg/Kg
				W-42271-110205-WA-24	6.8 U	µg/Kg

Notes:
(1) Results corrected for individual sample dilutions where applicable.
U Non-detect at associated value.

PROPRIETARY DOCUMENT

TABLE 10

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING LABORATORY CONTROL SAMPLE/LABORATORY CONTROL SAMPLE DUPLICATE RESULTS
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Compound*	*Preparation Date*	*LCS Percent Recovery*	*LCSD Percent Recovery*	*Control Limits (percent)*	*Associated Sample ID*	*Qualified Sample Results*	*Units*
VOCs in Air	1,2,4-Trichlorobenzene	11/10/05	41	48	70-130	A-42271-JC-001	0.50 UJ	ppbv
						A-42271-JC-002	0.50 UJ	ppbv
						A-42271-JC-003	1.5 UJ	ppbv
						A-42271-JC-004	2.5 UJ	ppbv
						A-42271-JC-005	1.5 UJ	ppbv
						A-42271-JC-006	0.50 UJ	ppbv
						A-42271-JC-007	0.50 UJ	ppbv
						A-42271-JC-008	5.0 UJ	ppbv
						A-42271-JC-009	5.0 UJ	ppbv
						A-42271-JC-010	0.50 UJ	ppbv
						A-42271-JC-011	0.50 UJ	ppbv
						A-42271-JC-012	5.0 UJ	ppbv
						A-42271-JC-013	2.5 UJ	ppbv
						A-42271-JC-014	0.50 UJ	ppbv
						A-42271-JC-015	1.5 UJ	ppbv
						A-42271-JC-017	5.0 UJ	ppbv
						A-42271-JC-018	1.1 UJ	ppbv
						A-42271-JC-019	0.50 UJ	ppbv
						A-42271-JC-020	0.50 UJ	ppbv
VOCs in Air	1,2,4-Trichlorobenzene	11/11/05	56	62	70-130	A-42271-JC-016	21 UJ	ppbv

Notes:
LCS Laboratory Control Sample.
LCSD Laboratory Control Sample Duplicate.
UJ The analyte was not detected above the sample quantitation limit. The reported quantitation limit is an estimated quantity.
VOCs Volatile Organic Compounds.

PROPRIETARY DOCUMENT

TABLE 11

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERIES
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Analyte*	*MS Recovery (percent)*	*MSD Recovery (percent)*	*RPD*	*Control Limits*		*Associated Sample ID*	*Qualified Sample Result*	*Units*
					Recovery (percent)	*RPD (percent)*			
Volatiles	Chlorobenzene	88	217	51	49-139	22	W-42271-110205-WA-22	50 J	µg/Kg
Metals	Lead	74	73	0.3	75-125	20	W-42271-110405-WA-08	1480 J	µg/L
							W-42271-110305-WA-01	40.7 J	µg/L
							W-42271-110305-WA-02	479 J	µg/L
							W-42271-110305-WA-03	102 J	µg/L
							W-42271-110405-WA-04	177 J	µg/L
							W-42271-110405-WA-05	1700 J	µg/L
							W-42271-110405-WA-06	28200 J	µg/L
							W-42271-110405-WA-07	26.2 J	µg/L
							W-42271-110405-WA-11	172 J	µg/L
							W-42271-110305-WA-03D	42.3	µg/L
Metals	Antimony	73	72	1.5	75-125	20	W-42271-110405-WA-08	6.6 J	µg/L
							W-42271-110305-WA-01	4.1 UJ	µg/L
							W-42271-110305-WA-02	6.7 J	µg/L
							W-42271-110305-WA-03	4.1 UJ	µg/L
							W-42271-110405-WA-04	4.1 UJ	µg/L
							W-42271-110405-WA-05	13.3 J	µg/L
							W-42271-110405-WA-06	197 J	µg/L
							W-42271-110405-WA-07	4.1 UJ	µg/L
							W-42271-110405-WA-11	7.5 J	µg/L
							W-42271-110305-WA-03D	4.1 UJ	µg/L
Metals	Calcium	71	73	0.61	75-125	20	W-42271-110405-WA-08	147000 J	µg/L
							W-42271-110305-WA-01	179000 J	µg/L
							W-42271-110305-WA-02	340000 J	µg/L
							W-42271-110405-WA-04	263000 J	µg/L

Notes:
J Estimated.
MS Matrix Spike.
MSD Matrix Spike Duplicate.
RPD Relative Percent Difference.
UJ The analyte was not detected above the sample quantitation limit. The reported quantitation limit is an estimated quantity.

PROPRIETARY DOCUMENT

TABLE 12

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING ICP SERIAL DILUTION RESULTS
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Analyte*	*Serial Dilution Sample ID*	*%D*	*Associated Sample I.D.*	*Qualified Sample Results*	*Units*
Metals	Nickel	W-42271-110305-WA-03D	20.4	W-42271-110305-WA-03D	71.1 J	µg/L
	Chromium	W-42271-110305-WA-03D	11.8	W-42271-110305-WA-03D	68.4 J	µg/L
	Cobalt	W-42271-110305-WA-03D	11.8	W-42271-110305-WA-03D	31.2 J	µg/L
	Copper	W-42271-110305-WA-03D	10.3	W-42271-110305-WA-03D	71.9 J	µg/L
	Zinc	W-42271-110305-WA-03D	16.5	W-42271-110305-WA-03D	187 J	µg/L
Metals	Zinc	W-42271-102505-WA-01	20.6	W-42271-102505-WA-01	41.7 J	mg/Kg
				S-42271-102705-WA-09-D	170 J	mg/Kg
				S-42271-102705-WA-10-D	99.0 J	mg/Kg
				S-042271-102705-WA-09	92.1 J	mg/Kg
				S-42271-102605-WA-02	108 J	mg/Kg
				S-42271-102605-WA-03	465 J	mg/Kg
				S-42271-102605-WA-04	176 J	mg/Kg
				S-42271-102605-WA-06	66.0 J	mg/Kg
				S-42271-102605-WA-07	836 J	mg/Kg
				S-42271-102605-WA-08	145 J	mg/Kg
				S-42271-102705-WA-10	84.1 J	mg/Kg
				S-42271-102705-WA-11	138 J	mg/Kg

Notes:
%D Percent Difference.
J Estimated.

PROPRIETARY DOCUMENT

TABLE 13

QUALIFIED SAMPLE RESULTS DUE TO VARIABILITY IN FIELD DUPLICATE RESULTS
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Analyte*	*Original Sample ID*	*Original Result*	*Duplicate Sample ID*	*Duplicate Result*	*RPD*	*Units*	*Qualifier* [1]
Volatiles	1,2-Dichlorobenzene	S-42271-102705-WA-10	6.2 U	S-42271-102705-WA-10-D	27 J	*	µg/Kg	J
	1,4-Dichlorobenzene	S-42271-102705-WA-10	6.2 U	S-42271-102705-WA-10-D	200 J	*	µg/Kg	J
	2-Butanone (Methyl Ethyl Ketone)	S-42271-102705-WA-10	4.5 J	S-42271-102705-WA-10-D	1200 U	*	µg/Kg	J
	Acetone	S-42271-102705-WA-10	200	S-42271-102705-WA-10-D	1200 U	*	µg/Kg	J
	Benzene	S-42271-102705-WA-10	20	S-42271-102705-WA-10-D	1900	196	µg/Kg	J
	Carbon disulfide	S-42271-102705-WA-10	2.3 J	S-42271-102705-WA-10-D	310 U	*	µg/Kg	J
	Chlorobenzene	S-42271-102705-WA-10	1.3 J	S-42271-102705-WA-10-D	130 J	196	µg/Kg	J
	cis-1,2-Dichloroethene	S-42271-102705-WA-10	0.96 J	S-42271-102705-WA-10-D	64 J	194	µg/Kg	J
	Cyclohexane	S-42271-102705-WA-10	12 U	S-42271-102705-WA-10-D	98 J	*	µg/Kg	J
	Ethylbenzene	S-42271-102705-WA-10	2.1 J	S-42271-102705-WA-10-D	380	198	µg/Kg	J
	Isopropylbenzene	S-42271-102705-WA-10	4.5 J	S-42271-102705-WA-10-D	600	197	µg/Kg	J
	Methyl cyclohexane	S-42271-102705-WA-10	1.1 J	S-42271-102705-WA-10-D	260 J	198	µg/Kg	J
	Methylene chloride	S-42271-102705-WA-10	13	S-42271-102705-WA-10-D	310 U	*	µg/Kg	J
	Toluene	S-42271-102705-WA-10	2.1 J	S-42271-102705-WA-10-D	230 J	196	µg/Kg	J
	Trichloroethene	S-42271-102705-WA-10	6.2 U	S-42271-102705-WA-10-D	18 J	*	µg/Kg	J
	Xylene (total)	S-42271-102705-WA-10	17	S-42271-102705-WA-10-D	2900	198	µg/Kg	J
Metals	Aluminum	W-42271-110305-WA-03	104000	W-42271-110305-WA-03D	48700	72	ug/L	J
	Arsenic	W-42271-110305-WA-03	46.7	W-42271-110305-WA-03D	18.3	87	ug/L	J
	Calcium	W-42271-110305-WA-03	819000	W-42271-110305-WA-03D	304000	92	ug/L	J
	Chromium	W-42271-110305-WA-03	164	W-42271-110305-WA-03D	68.4	82	ug/L	J
	Cobalt	W-42271-110305-WA-03	77.9	W-42271-110305-WA-03D	31.2	86	ug/L	J
	Copper	W-42271-110305-WA-03	193	W-42271-110305-WA-03D	71.9	91	ug/L	J
	Iron	W-42271-110305-WA-03	193000	W-42271-110305-WA-03D	77600	85	ug/L	J
	Lead	W-42271-110305-WA-03	102	W-42271-110305-WA-03D	42.3	83	ug/L	J
	Magnesium	W-42271-110305-WA-03	200000	W-42271-110305-WA-03D	96600	70	ug/L	J
	Manganese	W-42271-110305-WA-03	5880	W-42271-110305-WA-03D	2310	87	ug/L	J
	Nickel	W-42271-110305-WA-03	178	W-42271-110305-WA-03D	71.1	86	ug/L	J
	Vanadium	W-42271-110305-WA-03	235	W-42271-110305-WA-03D	99.6	81	ug/L	J
	Zinc	W-42271-110305-WA-03	465	W-42271-110305-WA-03D	187	85	ug/L	J

Notes:

* Cannot be calculated due to non-detect value.
(1) Qualifier is associated witih both the original and duplicate sample.
J Estimated.
RPD Relative Percent Difference.
U Non-detect at associated value.

TABLE 14

QUALIFIED SAMPLE RESULTS DUE TO ANALYTE CONCENTRATIONS IN THE RINSE BLANKS
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Rinse Blank Date*	*Analyte*	*Blank Result* [1]	*Sample ID*	*Qualified Sample Result*	*Units*
Volatiles	10/27/05	Acetone	3.6J	S-42271-102705-WA-09-D	31 U	µg/Kg
				S-42271-102705-WA-13	22 U	µg/Kg
Volatiles	11/03/05	Acetone	1.0J	W-42271-110305-WA-01	10 U	µg/L
				W-42271-110305-WA-02	10 U	µg/L
				W-42271-110305-WA-03	10 U	µg/L
General Chemistry	11/03/05	Cyanide	0.0028	SW-42271-110405-WA-09	0.010 U	mg/L
				W-42271-110305-WA-01	0.010 U	mg/L
				W-42271-110305-WA-02	0.010 U	mg/L
				W-42271-110305-WA-03	0.010 U	mg/L
				W-42271-110405-WA-04	0.010 U	mg/L
				W-42271-110405-WA-06	0.010 U	mg/L
				W-42271-110405-WA-07	0.010 U	mg/L
				W-42271-110405-WA-12	0.010 U	mg/L
				W-42271-110305-WA-03D	0.010 U	mg/L
Metals	10/27/05	Selenium	0.42	W-42271-102505-WA-01	1.7 U	mg/Kg
			0.48	W-42271-102805-WA-14	0.86 U	mg/Kg
			0.42	S-42271-102705-WA-09-D	1.3 U	mg/Kg
			0.38	S-42271-102705-WA-10-D	0.82 U	mg/Kg
			0.41	S-042271-102705-WA-09	0.54 U	mg/Kg
			0.38	S-42271-102605-WA-02	1.2 U	mg/Kg
			0.44	S-42271-102605-WA-03	0.91 U	mg/Kg
			0.38	S-42271-102605-WA-06	0.67 U	mg/Kg
			0.38	S-42271-102605-WA-07	1.3 U	mg/Kg
			0.38	S-42271-102705-WA-10	1.3 U	mg/Kg
			0.38	S-42271-103105-WA-16	1.2 U	mg/Kg

Notes:

(1) Blank results corrected for preparation factors and sample dilutions, where applicable.

U Non-detect at associated value.

PROPRIETARY DOCUMENT

TABLE 15

QUALIFIED SAMPLE RESULTS DUE TO ANALYTE CONCENTRATIONS IN THE TRIP BLANK
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Blank Date*	*Analyte*	*Blank Result*	*Associated Sample ID*	*Qualified Sample Result*	*Units*
Volatiles	10/27/05	Acetone	3.6J	S-42271-102705-WA-09-D	31 U	µg/Kg
				S-42271-102705-WA-13	22 U	µg/Kg
Volatiles	11/03/05	Acetone	0.97J	W-42271-110305-WA-01	10 U	µg/L
				W-42271-110305-WA-02	10 U	µg/L
				W-42271-110305-WA-03	10 U	µg/L

Notes:
J Estimated.
U Not detected.

TABLE 16

QUALIFIED SAMPLE RESULTS DUE TO DIFFERENCES IN DUAL COLUMN RESULTS
SUPPLEMENTAL REMEDIAL INVESTIGATION

Parameter	*Compound*	*Associated Sample ID*	*%D*	*Reported Results*	*Units*
Pesticides	gamma-Chlordane	S-42271-102605-WA-03	58.9	78J	µg/Kg
Pesticides	gamma-Chlordane	S-42271-102605-WA-04	68.6	84J	µg/Kg
Pesticides	Methoxychlor	S-42271-102605-WA-08	43.9	1100J	µg/Kg
Pesticides	Endrin ketone	S-42271-102605-WA-08	42.4	280J	µg/Kg

Notes:
%D Percent Difference.
J Estimated.

TABLE 17A

ANALYTICAL RESULTS SUMMARY - SOIL SAMPLING - TICs
SUPPLEMENTAL REMIEDIAL INVESTIGATION

	Sample Location:	*MW-3-05*	*MW-3-05*	*MW-5-05*	*MW-7-05*
	Sample ID:	*S-42271-102605-WA-06*	*S-42271-102605-WA-07*	*S-42271-102605-WA-08*	*S-42271-110105-WA-21*
	Sample Date	*10/26/2005*	*10/26/2005*	*10/26/2005*	*11/1/2005*
Parameter	*Units*				
Tentatively Identified Compounds (TICs) - Volatile					
1,2,3,4-Tetramethyl-benzene	µg/kg	--	--	--	--
1,2,3-Trimethylbenzene	µg/kg	12 NJ	--	--	--
1-ethyl-2,4-dimethylbenzene	µg/kg	--	--	--	--
1-Ethyl-2-methylbenzene	µg/kg	6.0 NJ	--	--	--
1-Ethyl-2-methyl-cyclohexane	µg/kg	--	--	--	--
1-Ethyl-3-methylbenzene	µg/kg	--	--	--	--
1-Methyl-2-prop-cyclohexane	µg/kg	--	17000 NJ	--	--
1-Propenylbenzene	µg/kg	--	--	--	--
4-Ethyl-1,2-dimethylbenzene	µg/kg	6.8 NJ	--	--	--
4-Ethyl-2,2,6,6-tetramethylheptane	µg/kg	--	--	--	--
4-Octene, 2,6-dimethyl-, [S	µg/kg	--	14000 NJ	--	--
Benzene, (1-methyl-1-propenyl)-, (E)-	µg/kg	--	--	--	--
Benzene, 1,2-diethyl-	µg/kg	9.3 NJ	--	--	--
Benzene, 1,3,5-trimethyl-	µg/kg	--	--	--	--
Benzene, 1-ethyl-3,5-dimethyl-	µg/kg	7.2 NJ	--	--	--
Benzene, 1-methyl-2-(1-methylethyl)-	µg/kg	7.8 NJ	--	--	--
Benzene, 1-methyl-3-(1-methylethyl)-	µg/kg	--	--	--	--
Benzene, 1-methylpropyl-	µg/kg	--	--	--	--
Benzene, 2-ethly-1,4-dimethy	µg/kg	--	--	--	--
Bicyclo[3.3.1]nonane	µg/kg	--	--	--	--

TABLE 17A

ANALYTICAL RESULTS SUMMARY - SOIL SAMPLING - TICs
SUPPLEMENTAL REMIEDIAL INVESTIGATION

	Sample Location:	*MW-3-05*	*MW-3-05*	*MW-5-05*	*MW-7-05*
	Sample ID:	*S-42271-102605-WA-06*	*S-42271-102605-WA-07*	*S-42271-102605-WA-08*	*S-42271-110105-WA-21*
	Sample Date	*10/26/2005*	*10/26/2005*	*10/26/2005*	*11/1/2005*
Parameter	*Units*				
Tentatively Identified Compounds (TICs) - Volatile (Cont'd.)					
Camphene	µg/kg	--	--	--	--
Cycloheptane, methyl-	µg/kg	--	--	--	--
Cyclohexane, 1-methyl-3-pro	µg/kg	--	--	--	--
Cyclohexane, 1-methyl-4-(1-	µg/kg	--	--	--	--
Cyclohexane, butyl-	µg/kg	--	14000 NJ	--	--
Cymene	µg/kg	6.4 NJ	--	--	--
Decahydro-naphthalene	µg/kg	--	--	--	--
Decane, 3-methyl-	µg/kg	--	--	--	--
Dimethyl sulfide	µg/kg	--	--	--	--
Heptane, 3,3,5-trimethyl-	µg/kg	--	8700 NJ	--	--
Isopropyltoluene	µg/kg	--	--	--	--
Naphthalene, decahydro-, trans-	µg/kg	--	--	--	--
Octane, 3,3-dimethyl-	µg/kg	--	--	--	--
Octane, 4-ethyl-	µg/kg	--	10000 NJ	--	--

TABLE 17A

ANALYTICAL RESULTS SUMMARY - SOIL SAMPLING - TICs
SUPPLEMENTAL REMIEDIAL INVESTIGATION

	Sample Location:	*MW-9-05*	*POND*	*Low lying area near RCR Yacht*
	Sample ID:	*S-42271-110105-WA-18*	*S-42271-110305-WA-027*	*S-42271-110305-WA-025*
	Sample Date	*11/1/2005*	*11/3/2005*	*11/3/2005*
Parameter	*Units*			
Tentatively Identified Compounds (TICs) - Volatile				
1,2,3,4-Tetramethyl-benzene	µg/kg	--	--	--
1,2,3-Trimethylbenzene	µg/kg	--	--	--
1-ethyl-2,4-dimethylbenzene	µg/kg	--	--	--
1-Ethyl-2-methylbenzene	µg/kg	--	--	--
1-Ethyl-2-methyl-cyclohexane	µg/kg	--	--	--
1-Ethyl-3-methylbenzene	µg/kg	--	--	--
1-Methyl-2-prop-cyclohexane	µg/kg	--	--	--
1-Propenylbenzene	µg/kg	--	--	--
4-Ethyl-1,2-dimethylbenzene	µg/kg	--	--	--
4-Ethyl-2,2,6,6-tetramethylheptane	µg/kg	--	--	--
4-Octene, 2,6-dimethyl-, [S	µg/kg	--	--	--
Benzene, (1-methyl-1-propenyl)-, (E)-	µg/kg	--	--	--
Benzene, 1,2-diethyl-	µg/kg	--	--	--
Benzene, 1,3,5-trimethyl-	µg/kg	--	--	--
Benzene, 1-ethyl-3,5-dimethyl-	µg/kg	--	--	--
Benzene, 1-methyl-2-(1-methylethyl)-	µg/kg	--	--	--
Benzene, 1-methyl-3-(1-methylethyl)-	µg/kg	--	--	--
Benzene, 1-methylpropyl-	µg/kg	--	--	--
Benzene, 2-ethly-1,4-dimethy	µg/kg	--	--	--
Bicyclo[3.3.1]nonane	µg/kg	--	--	--

TABLE 17A

ANALYTICAL RESULTS SUMMARY - SOIL SAMPLING - TICs
SUPPLEMENTAL REMIEDIAL INVESTIGATION

	Sample Location:	*MW-9-05*	*POND*	*Low lying area near RCR Yacht*
	Sample ID:	*S-42271-110105-WA-18*	*S-42271-110305-WA-027*	*S-42271-110305-WA-025*
	Sample Date	*11/1/2005*	*11/3/2005*	*11/3/2005*
Parameter	*Units*			
Tentatively Identified Compounds (TICs) - Volatile (Cont'd.)				
Camphene	µg/kg	--	--	--
Cycloheptane, methyl-	µg/kg	--	--	--
Cyclohexane, 1-methyl-3-pro	µg/kg	--	--	--
Cyclohexane, 1-methyl-4-(1-	µg/kg	--	--	--
Cyclohexane, butyl-	µg/kg	--	--	--
Cymene	µg/kg	--	--	--
Decahydro-naphthalene	µg/kg	--	--	--
Decane, 3-methyl-	µg/kg	--	--	--
Dimethyl sulfide	µg/kg	--	8.3 NJ	--
Heptane, 3,3,5-trimethyl-	µg/kg	--	--	--
Isopropyltoluene	µg/kg	--	--	--
Naphthalene, decahydro-, trans-	µg/kg	--	--	--
Octane, 3,3-dimethyl-	µg/kg	--	--	--
Octane, 4-ethyl-	µg/kg	--	--	--

Notes:

-	Not analyzed.
J	Estimated.
N	Tentatively Identified.
PAH	Polynuclear Aromatic Hydrocarbon.

APPENDIX B-2

FULL DATA VALIDATION MEMO TEMPLATE

CONESTOGA-ROVERS & ASSOCIATES

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PROPRIETARY DOCUMENT

MEMORANDUM

TO:

REF. NO.:

FROM:

DATE: February 12, 2008
E-Mail and Hard Copy if Requested

RE: **Analytical Results and QA/QC Review**
Groundwater and Soil Investigation

July - August 2007

The following details a quality assessment and validation of the analytical data resulting from the July and August 2007 collection of groundwater and soil samples from the _____ Site in _____. The sample summary detailing sample identification, sample location, quality control (QC) samples, and analytical parameters is presented in Table 1. Sample analysis was completed at _____Laboratories, in _____, in accordance with the methodologies presented in Table 2. Summaries of the analytical results are presented in Tables 3A and 3B.

Evaluation of the data was based on information obtained from the finished data sheets, raw data, Chain of Custody forms, calibration data, duplicate data and recovery for matrix, blank, and surrogate spikes. The assessment of analytical and in-house data included checks for: data consistency (by observing comparability of duplicate analyses); adherence to accuracy and precision criteria; transmittal errors; and anomalously high and low parameter values.

The QC criteria used to assess the data were established by the methods and with the following guidance documents:

i) "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", United States Environmental Protection Agency (USEPA) 540/R-99/008, October 1999; and

ii) "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Review", USEPA 540/R-94/013, February 1994.

These guidelines are collectively referred to as "Guidelines" in this memorandum.

Sample Quantitation

The laboratory reported detected concentrations of organic compounds and metals below the laboratory's practical quantitation limit (PQL)/report limit (RL) but above the laboratory's method detection limit (MDL). The laboratory flagged these sample concentrations with a "J". These concentrations should be qualified as estimated (J) values unless qualified otherwise in this memorandum.

Sample Preservation and Holding Times

Sample holding time periods and preservation requirements are summarized in the analytical methods. All sample extractions and/or analyses were performed within the specified holding times.

All samples were properly preserved and cooled to 4°C(±2°C) after collection.

Gas Chromatography/Mass Spectrometer (GC/MS) - Tuning and Mass Calibration (Instrument Performance Check) - Volatile Organic Compounds (VOCs) and Semi-Volatile Organic Compounds (SVOCs)

To ensure adequate mass resolution, identification, and to some degree, sensitivity; the performance of each GC/MS instrument used for VOC and SVOC analyses was checked at the beginning of each 12-hour period using bromofluorobenzene (BFB) and decafluorotriphenylphosphine (DFTPP), respectively. The resulting spectra must meet the criteria cited in the "Guidelines" before initiating an analysis sequence.

Instrument performance check data were reviewed. These tuning compounds were analyzed at the required frequency throughout the VOC and SVOC analyses. The results of all instrument performance checks were within the acceptance criteria, indicating acceptable instrument performance.

GC/MS Initial Calibration - VOCs and SVOCs

Initial calibration data are used to demonstrate that each instrument is capable of generating acceptable quantitative data. A five point calibration curve containing all compounds of interest is analyzed to characterize instrument response for each over a specific concentration range.

Initial calibration criteria for organic analyses are evaluated against the following criteria:

i) must meet a minimum mean relative response factor (RRF) of 0.05; and

ii) the percent relative standard deviation (%RSD) values must not exceed 30.0 percent or a minimum coefficient of determination (R^2) of 0.99 if quadratic equation calibration curves are used.

Calibration standards were analyzed at the required frequency and the results met the above criteria for sensitivity. Some SVOC compounds exhibited a high standard deviation, therefore not meeting the linearity criteria. All associated results were qualified as estimated (see Table 4).

GC/MS Continuing Calibration - VOCs and SVOCs

To ensure that each instrument was capable of producing acceptable quantitative data over the analysis period, continuing calibration standards must be analyzed every 12 hours. The following criteria are employed to evaluate the continuing calibration data:

i) must meet a minimum mean RRF of 0.05;

ii) the percent difference (%D) between the mean initial calibration RRF and the continuing calibration RRF must not exceed 25 percent; and

iii) the percent drift between the true value and the continuing calibration value must not exceed 25 percent.

Calibration standards were analyzed at the required frequency and the results met the above criteria for instrument sensitivity and linearity of response for most analytes. Some VOCs and SVOCs exhibited variability between the initial and continuing response factors. All associated results were qualified as estimated (see Table 5).

GC Initial Calibration – Pesticides, Herbicides, and Polychlorinated Biphenyls (PCBs)

To quantify compounds of interest, calibration of the GC over a specific concentration range must be performed. Initially, five-point calibration curves are analyzed for all the pesticides and herbicides of interest. PCB calibration curves are analyzed for Aroclors 1016 and1260 only. A single point calibration is performed for the remaining Aroclors of interest.

Linearity of the calibration curves are acceptable if percent %RSD values are less than or equal to 20 percent or if the coefficient of determination (R^2) is greater than 0.99. Retention time (RT) windows are also calculated from the initial calibration analyses. These windows are then used to identify all compounds of interest in subsequent analyses.

Initial calibration standards were analyzed at the required frequencies. All RT and linearity criteria were satisfied.

GC Continuing Calibration – Pesticides, Herbicides and PCBs

To ensure that the calibration of the instrument is valid throughout the sample analysis period, continuing calibration standards are analyzed and evaluated on a regular basis. To evaluate the continued linearity of the calibration, %D values are calculated for each compound in all continuing standards and assessed against an acceptance criterion of 15 percent.

To ensure that compound RTs do not vary over the analysis period, all RTs must fall within the established RT windows.

Continuing calibration standards were analyzed at the required frequency, and all method criteria were met for analyte linearity with the exception of some pesticides and herbicides exhibiting a %D greater than 15. All associated sample results were qualified as estimated (see Table 5).

Initial Calibration - Inorganic Analyses

Initial calibration of the instruments ensures that they are capable of producing satisfactory quantitative data at the beginning of a series of analyses. For trace inductively coupled plasma (ICP) analysis, a calibration blank and at least one standard must be analyzed at each wavelength to establish the analytical curve. Mercury analysis by cold vapor atomic absorption spectroscopy (CVAA) requires the analysis of a calibration blank and a minimum of five standards to establish the calibration curve. The coefficient of variation for calibration curves must exceed 0.995.

Initial calibration is verified with an initial calibration verification (ICV) standard which must recover within 90 to 110 percent for metals by ICP and 80 to 120 percent for mercury by CVAA.

A review of the laboratory data showed that all inorganic initial calibration curves and ICVs were analyzed at the appropriate frequency and were within the acceptance criteria.

Continuing Calibration - Inorganic Analyses

Continuing calibration verification (CCV) standards are analyzed at method specified frequency (one every 10 samples). The CCVs must meet the percent recovery control limits specified above for the ICVs. Criteria for inorganic analyses are the same criteria as used for assessing the initial calibration data.

A review of the laboratory data showed that CCVs were analyzed at the appropriate frequency and the data were within the acceptance criteria.

Method Blank Samples

Method blank samples are prepared from a purified sample matrix and are processed concurrently with investigative samples to assess the presence and the magnitude of sample contamination introduced during sample analysis. Method blank samples are analyzed at a minimum frequency of one per analytical batch and target analytes should be non-detect.

Method blanks were analyzed at the recommended frequency and the results were non-detect for all analytes of interest with the exception of methylene chloride and various metals present at low level concentrations. All associated positive sample results with similar concentrations were qualified as non-detect (see Table 6).

Laboratory Blank Samples - Inorganic Analyses

Metals analyses include the analysis of initial calibration blanks (ICB) and continuing calibration blanks (CCB) to assess the presence and the magnitude of sample contamination introduced during sample analysis. The CCBs are analyzed at a minimum frequency of one every 10 samples and target analytes should be non-detect.

All ICBs and CCBs were non-detect with the exception of beryllium present on September 7, 2007. All associated positive sample results with similar concentrations were qualified as non-detect (see Table 6).

Surrogate Compounds - Organic Analyses

Individual sample performance for organic analyses was monitored by assessing the results of surrogate compound percent recoveries. Surrogate percent recoveries are reviewed against the laboratory developed control limits provided in the analytical report.

All surrogate recoveries met the method criteria, demonstrating acceptable analytical efficiency for these analyses with the exception of some high SVOC surrogate recoveries. All associated positive sample results were qualified as estimated (see Table 7) and all non-detect results would not have been impacted by the implied high bias.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

To assess the long term accuracy and precision of the analytical methods on various matrices, MS/MSD percent recoveries and relative percent differences (RPD) of the concentrations were determined. The organic MS/MSD percent recovery and RPD control limits are established by the laboratory. The inorganic control limits are defined by the methods and the "Guidelines", which require recoveries between 75 to 125 percent with RPDs less than 20 percent.

All MS/MSD recoveries were acceptable with the following exceptions:

i) high aluminum and cyanide recoveries were observed. All associated positive results were qualified as estimated and all non-detect results would not have been impacted by the implied high bias;

ii) due to matrix interference, the 4,4'-dichlorodiphenyltrichloroethane (DDT) recoveries could not be calculated. The associated sample result was qualified as estimated; and

iii) high variability was observed between trichloroethene recoveries. The associated sample result was qualified as estimated.

A summary of the qualified dated is presented in Table 8.

Laboratory Control Sample (LCS)

The LCS analysis serves as a monitor of the overall performance in all steps of the sample analysis and are analyzed with each sample batch. The LCS percent recoveries were evaluated against method and laboratory established control limits. Some LCS analyses were performed in duplicate to monitor laboratory precision.

The LCS percent recoveries were all within the laboratory control limits indicating acceptable analytical accuracy and precision (where applicable).

Internal Standard (IS) Summaries - Organic Analyses

To correct for variability in the GC/MS response and sensitivity, IS compounds are added to all samples. All results are calculated as a ratio of the compound and associated IS response. Overall instrument stability and performance for VOC and SVOC analyses were monitored using IS peak area and RT data. The IS peak areas and RTs of the samples are required to meet the following criteria:

i) IS area counts must not vary by more than a factor of two (-50 percent to +100 percent) from the associated continuing calibration standard IS area counts; and

ii) the RT of the IS must not vary by more than plus or minus 30 seconds from the associated continuing calibration standard.

A review of the VOC and SVOC internal standard data showed that the IS area counts and RT data were within the acceptance criteria for all VOC and SVOC samples.

ICP ICS Analysis - Inorganic Analyses

To verify that proper inter-element and background correction factors had been established by the laboratory for metals analyses, the ICP ICS are analyzed. The ICSs are evaluated against recovery control limits of 80 to 120 percent.

The ICS analysis results were evaluated for all samples and were within the control limits.

Serial Dilution - Inorganic Analyses

The %D between a serial dilution of a sample for each matrix was monitored to determine physical or chemical interference. A minimum of one sample per 20 investigative samples is analyzed at a five-fold dilution. The serial dilution results must agree within 10 %D of the original results for samples with detected concentrations greater than 50 times the instrument detection limit.

The %D acceptance criteria was met for all metals.

Target Compound Identification

To minimize erroneous compound identification during organic analyses, qualitative criteria including compound RT and mass spectra (if applicable) were evaluated according to identification criteria established by the methods. The organic compounds reported adhered to the specified identification criteria.

Target Compound Quantitation

The reported quantitation results and detection limits were checked to ensure results reported were accurate. No discrepancies were found between the raw data and the sample results reported by the laboratory.

Field Quality Assurance/Quality Control (QA/QC)

The field QA/QC consisted of five field duplicate sample sets and a trip blank.

Overall precision for the sampling event and laboratory procedures was monitored using the results of the field duplicate sample sets. The RPDs associated with these duplicate samples must be less than 50 percent for water and 100 percent for soil/sediment. If the reported concentration in either the investigative sample or its duplicate is less than five times the RL, the evaluation criteria is one times the RL value for water or two times for soil/sediment.

Most field duplicate results were acceptable indicating good field and analytical precision. Some variability was observed between VOC and metals concentrations. The samples and the associated field duplicate were qualified as estimated to reflect the implied variability (see Table 9).

To monitor potential cross-contamination of VOC during aqueous sample transportation and storage, a trip blank was submitted to the laboratory for VOC analysis with each shipping cooler containing multiple samples.

All trip blank results were non-detect for the compounds of interest.

Dual Column Analysis

Pesticide analyses were performed using dual column analyses. In general, the pesticide results showed good correlation between the two columns. Variability was observed between some of the results (see Table 10). The associated data were qualified as estimated to reflect the implied variability.

System Performance

System performance between various quality control checks was evaluated to monitor for changes that may have caused the degradation of data quality. No technical problems or chromatographic anomalies were observed which would require qualification of the data.

Overall Assessment

The data were found to exhibit acceptable levels of accuracy and precision, based on the provided information, and may be used with the qualifications noted herein.

PROPRIETARY DOCUMENT

TABLE 1

SAMPLE COLLECTION AND ANALYSIS SUMMARY
GROUNDWATER AND SOIL INVESTIGATION
SITE
JULY - AUGUST 2007

						Analysis/Parameters									
Sample I.D.	*Location I.D.*	*Collection Date (mm/dd/yy)*	*Collection Time (hr:min)*	*Start Depth (ft bgs)*	*End Depth (ft bgs)*	*TCL VOCs*	*TCL SVOCs*	*Lead*	*TAL Metals + Cn*	*Pesticides*	*Herbicides*	*PCBs*	*Dissolved Metals*	*Diss. Lead*	*Comments*
SO-37191-072507-RN-SB-1	SB-1-07	07/25/07	10:11	2	4	X	X		X	X	X	X			
SO-37191-072507-RN-SB-05	SB-5-07	07/25/07	12:44	4	8	X	X		X	X	X	X			
SO-37191-072507-RN-SB-10	SB-10-07	07/25/07	13:24	3	8	X	X	X							
SO-37191-072507-RN-SB-9	SB-9-07	07/25/07	13:50	3	6	X	X	X							
SO-37191-072507-RN-SB-7	SB-7-07	07/25/07	14:21	3	6	X	X	X							
SO-37191-072507-RN-SB-8	SB-8-07	07/25/07	14:55	3.5	8	X	X		X	X	X	X			
SO-37191-072607-RN-SB-11	SB-11-07	07/26/07	8:53	2	6	X	X	X							
SO-37191-072607-RN-SB-15	SB-15-07	07/26/07	9:33	4	8	X	X	X							

Notes:

-	Not analyzed.
Cn	Cyanide.
PCBs	Polychlorinated Biphenyls.
SVOCs	Semi-Volatile Organic Compounds.
TAL	Target Analyte List.
TCL	Target Compound List.
VOCs	Volatile Organic Compounds.

PROPRIETARY DOCUMENT

TABLE 2

SUMMARY OF ANALYTICAL METHODS
GROUNDWATER AND SOIL SAMPLING
SITE
JULY - AUGUST 2007

Parameter	*Method* [1]
Soil	
TCL VOCs	SW-846 8260B
TCL SVOCs	SW-846 8270C
TCL Pesticides	SW-846 8081
Herbicides	SW-846 8151
Polychlorinated Biphenyls	SW-846 8082
TAL Metals	SW-846 6010/7000 Series
Cyanide	SW-846 9012
Groundwater	
TCL VOCs	SW-846 8260B
TCL SVOCs	SW-846 8270C
TCL Pesticides	SW-846 8081
Herbicides	SW-846 8151
Polychlorinated Biphenyls	SW-846 8082
TAL Metals (total and dissolved)	SW-846 6010/7000 Series
Cyanide (total and dissolved)	SW-846 9012

Notes:

[1] "Test Methods for Solid Waste/Physical Chemical Methods", SW-846, 3rd Edition, September 1986 (with all subsequent revisions).

SVOCs Semi-Volatile Organic Compounds.

TAL Target Analyte List.

TCL Target Compound List.

VOCs Volatile Organic Compounds.

TABLE 3A
ANALYTICAL RESULTS SUMMARY
GROUNDWATER AND SOIL INVESTIGATION
SITE
JULY-AUGUST 2007

			Location ID:	*MW-5/BH-5*	*MW-8/BH-15*	*MW-9/BH-1*	*MW-12*
			Sample Name:	*WG-37191-082107-RN-003*	*WG-37191-082107-RN-006*	*WG-37191-082107-RN-007*	*WG-37191-082107-RN-004*
			Sample Date:	*8/21/2007*	*8/21/2007*	*8/21/2007*	*8/21/2007*
		New York State Water Quality					
Parameters	*Units*	*Standards*	*Guidance Values*				
Volatile Organic Compounds							
1,1,1-Trichloroethane	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
1,1,2,2-Tetrachloroethane	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
1,1,2-Trichloroethane	ug/L	1	NC	9800 J	5.0 U	5.0 U	15000 U
1,1-Dichloroethane	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
1,1-Dichloroethene	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
1,2,4-Trichlorobenzene	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
1,2-Dibromo-3-chloropropane (DBCP)	ug/L	0.04	NC	12000 U	5.0 U	5.0 U	15000 U
1,2-Dibromoethane (Ethylene Dibromide)	ug/L	0.0006	NC	12000 U	5.0 U	5.0 U	15000 U
1,2-Dichlorobenzene	ug/L	3	NC	12000 U	5.0 U	5.0 U	15000 U
1,2-Dichloroethane	ug/L	0.6	NC	12000 U	5.0 U	5.0 U	15000 U
1,2-Dichloropropane	ug/L	1	NC	12000 U	5.0 U	5.0 U	15000 U
1,3-Dichlorobenzene	ug/L	3	NC	12000 U	5.0 U	5.0 U	15000 U
1,4-Dichlorobenzene	ug/L	3	NC	12000 U	5.0 U	5.0 U	15000 U
2-Butanone (Methyl Ethyl Ketone)	ug/L	50	50	12000 U	5.0 U	5.0 U	15000 U
2-Hexanone	ug/L	50	50	12000 U	5.0 U	5.0 U	15000 U
4-Methyl-2-Pentanone (Methyl Isobutyl Ketone)	ug/L	NC	NC	12000 U	5.0 U	5.0 U	15000 U
Acetone	ug/L	NC	50	50000 U	20 UJ	20 UJ	60000 UJ
Benzene	ug/L	1	NC	12000 U	5.0 U	5.0 U	15000 U
Bromodichloromethane	ug/L	50	50	12000 U	5.0 U	5.0 U	15000 U
Bromoform	ug/L	50	50	12000 U	5.0 U	5.0 U	15000 U
Bromomethane (Methyl Bromide)	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
Carbon disulfide	ug/L	NC	60	12000 U	5.0 U	5.0 U	15000 U
Carbon tetrachloride	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
Chlorobenzene	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
Chloroethane	ug/L	5	NC	12000 UJ	5.0 U	5.0 U	15000 U
Chloroform (Trichloromethane)	ug/L	7	NC	12000 U	5.0 U	5.0 U	15000 U
Chloromethane (Methyl Chloride)	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
cis-1,2-Dichloroethene	ug/L	5	NC	240000	1.0 J	5.0 U	65000 J
cis-1,3-Dichloropropene	ug/L	NC	NC	12000 U	5.0 U	5.0 U	15000 U
Cyclohexane	ug/L	NC	NC	12000 U	5.0 U	5.0 U	15000 U
Dibromochloromethane	ug/L	50	50	12000 U	5.0 U	5.0 U	15000 U
Dichlorodifluoromethane (CFC-12)	ug/L	5	NC	12000 U	5.0 UJ	5.0 UJ	15000 UJ
Ethylbenzene	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
Isopropylbenzene	ug/L	5	NC	12000 U	5.0 U	5.0 U	15000 U
Methyl acetate	ug/L	NC	NC	12000 U	5.0 U	5.0 U	15000 U
Methyl cyclohexane	ug/L	NC	NC	12000 U	5.0 U	5.0 U	15000 U
Methyl Tert Butyl Ether	ug/L	NC	10	12000 U	5.0 U	5.0 U	15000 U

TABLE 3A
ANALYTICAL RESULTS SUMMARY
GROUNDWATER AND SOIL INVESTIGATION
SITE
JULY-AUGUST 2007

			Location ID:	*MW-5/BH-5*	*MW-8/BH-15*	*MW-9/BH-1*	*MW-12*
			Sample Name:	*WG-37191-082107-RN-003*	*WG-37191-082107-RN-006*	*WG-37191-082107-RN-007*	*WG-37191-082107-RN-004*
			Sample Date:	*8/21/2007*	*8/21/2007*	*8/21/2007*	*8/21/2007*
		New York State Water Quality					
Parameters	*Units*	*Standards*	*Guidance Values*				
PCBs							
Aroclor-1016 (PCB-1016)	ug/L	NC	NC	–	–	0.41 U	–
Aroclor-1221 (PCB-1221)	ug/L	NC	NC	–	–	0.41 U	–
Aroclor-1232 (PCB-1232)	ug/L	NC	NC	–	–	0.41 U	–
Aroclor-1242 (PCB-1242)	ug/L	NC	NC	–	–	0.41 U	–
Aroclor-1248 (PCB-1248)	ug/L	NC	NC	–	–	0.41 U	–
Aroclor-1254 (PCB-1254)	ug/L	NC	NC	–	–	0.41 U	–
Aroclor-1260 (PCB-1260)	ug/L	NC	NC	–	–	0.41 U	–
WetChemistry							
Cyanide (total)	ug/L	200	NC	–	–	2.4 J	–

Notes:

-	Not analyzed.
1.0	Exceeds Criteria.
B	Compound detected in an associated blank.
BHC	Benzene Hexachloride.
D	Reported from a diluted analysis.
E	Exceeds the linear range of the instrument.
GC	Gas Chromatograph
J	Estimated.
N	Tentatively identified.
NC	No Criteria.
P	Greater
PCBs	Polychlorinated Biphenyls.
U	Not detected.
UJ	Not detected, estimated reporting limit.

TABLE 3B

ANALYTICAL RESULTS SUMMARY
SOIL INVESTIGATION
SITE
JULY - AUGUST 2007

		Location ID:	*SB-1-07*	*SB-2-07*	*SB-2-07*	*SB-3-07*	*SB-4-07*
		Sample Name:	*SO-37191-072507-RN-SB-1*	*SO-37191-072707-RN-SB-2*	*SO-37191-072707-RN-SB-20*	*SO-37191-072707-RN-SB-3*	*SO-37191-072707-RN-SB-4*
		Sample Date:	*7/25/2007*	*7/27/2007*	*7/27/2007*	*7/27/2007*	*7/27/2007*
		Depth:	*2 - 4 ft*	*6.5 - 8 ft*	*6.5 - 8 ft*	*10 - 13 ft*	*2 - 4 ft*
					Duplicate		
Parameters	*Units* [1]	*6 NYCRR Part 375-6.8(b): Restricted Use Soil Cleanup Objectives Protection of Public Health - Industrial*					
Volatile Organic Compounds							
1,1,1-Trichloroethane	mg/kg	1000	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,1,2,2-Tetrachloroethane	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,1,2-Trichloroethane	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,1-Dichloroethane	mg/kg	480	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,1-Dichloroethene	mg/kg	1000	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,2,4-Trichlorobenzene	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,2-Dibromo-3-chloropropane (DBCP)	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,2-Dibromoethane (Ethylene Dibromide)	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,2-Dichlorobenzene	mg/kg	1000	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,2-Dichloroethane	mg/kg	60	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,2-Dichloropropane	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,3-Dichlorobenzene	mg/kg	560	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
1,4-Dichlorobenzene	mg/kg	250	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
2-Butanone (Methyl Ethyl Ketone)	mg/kg	1000	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
2-Hexanone	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
4-Methyl-2-Pentanone (Methyl Isobutyl Ketone)	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Acetone	mg/kg	1000	0.028 U	0.025 UJ	0.023 J	0.024 UJ	0.025 U
Benzene	mg/kg	89	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Bromodichloromethane	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Bromoform	mg/kg	NC	0.0069 UJ	0.0062 UJ	0.0067 UJ	0.006 UJ	0.0062 UJ
Bromomethane (Methyl Bromide)	mg/kg	NC	0.0069 UJ	0.0062 UJ	0.0067 UJ	0.006 UJ	0.0062 UJ
Carbon disulfide	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Carbon tetrachloride	mg/kg	44	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Chlorobenzene	mg/kg	1000	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Chloroethane	mg/kg	NC	0.0069 UJ	0.0062 UJ	0.0067 UJ	0.006 UJ	0.0062 UJ
Chloroform (Trichloromethane)	mg/kg	700	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Chloromethane (Methyl Chloride)	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
cis-1,2-Dichloroethene	mg/kg	1000	0.0069 U	0.0014 J	0.0067 U	0.006 U	0.0062 U
cis-1,3-Dichloropropene	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Cyclohexane	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Dibromochloromethane	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Dichlorodifluoromethane (CFC-12)	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U

TABLE 3B

ANALYTICAL RESULTS SUMMARY
SOIL INVESTIGATION
SITE
JULY - AUGUST 2007

		Location ID:	*SB-1-07*	*SB-2-07*	*SB-2-07*	*SB-3-07*	*SB-4-07*
		Sample Name:	*SO-37191-072507-RN-SB-1*	*SO-37191-072707-RN-SB-2*	*SO-37191-072707-RN-SB-20*	*SO-37191-072707-RN-SB-3*	*SO-37191-072707-RN-SB-4*
		Sample Date:	*7/25/2007*	*7/27/2007*	*7/27/2007*	*7/27/2007*	*7/27/2007*
		Depth:	*2 - 4 ft*	*6.5 - 8 ft*	*6.5 - 8 ft*	*10 - 13 ft*	*2 - 4 ft*
					Duplicate		
Parameters	*Units* [1]	*6 NYCRR Part 375-6.8(b): Restricted Use Soil Cleanup Objectives Protection of Public Health - Industrial*					
Ethylbenzene	mg/kg	780	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Isopropylbenzene	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Methyl acetate	mg/kg	NC	0.0069 U	0.0062 U	0.0067 U	0.006 U	0.0062 U
Pesticides (Cont'd.)							
Methoxychlor	mg/kg	NC	0.0035 J	–	–	0.0039 U	–
Toxaphene	mg/kg	NC	0.093 U	–	–	0.079 U	–
PCBs							
Aroclor-1016 (PCB-1016)	mg/kg	25	0.023 U	–	–	0.02 U	–
Aroclor-1221 (PCB-1221)	mg/kg	25	0.023 U	–	–	0.02 U	–
Aroclor-1232 (PCB-1232)	mg/kg	25	0.023 U	–	–	0.02 U	–
Aroclor-1242 (PCB-1242)	mg/kg	25	0.023 U	–	–	0.02 U	–
Aroclor-1248 (PCB-1248)	mg/kg	25	0.023 U	–	–	0.02 U	–
Aroclor-1254 (PCB-1254)	mg/kg	25	0.03	–	–	0.02 U	–
Aroclor-1260 (PCB-1260)	mg/kg	25	0.018 J	–	–	0.02 U	–
Wet Chemistry							
Cyanide (total)	mg/kg	10000	0.29 J	–	–	0.60 U	–
Total Solids	%	NC	72.4	80.7	75.2	83.8	81

Notes:

[1] Reported results were converted from ug/kg (ppb) to mg/kg (ppm) for ease of comparison to criteria.

[2] The Soil Cleanup Objective (SCO) for this specific compound (or family of compounds) is considered to be met if the analysis for the total species of this contaminant is below the specific SCO. The most restrictive SCO for hexavalent Chromium was used for comparison to the total chromium results.

- Not analyzed.

1.0 Exceeds Criteria.

B Compound detected in an associated blank.

BHC Benzene Hexachloride.

D Reported from a diluted analysis.

E Exceeds the linear range of the instrument.

GC Gas Chromatograph

J Estimated.

N Tentatively identified.

NC No Criteria.

P Greater than 25% difference between concentrations detected on the two GC columns.

PCBs Polychlorinated Biphenyls.

U Not detected.

UJ Not detected, estimated reporting limit.

PROPRIETARY DOCUMENT

TABLE 4

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING INITIAL CALIBRATION RESULTS
GROUNDWATER AND SOIL SAMPLING
SITE
JULY - AUGUST 2007

Parameter	*Compound*	*Calibration Date*	*%RSD*	*Associated Sample ID*	*Qualified Sample Results*	*Units*
SVOCs	2,4-Dinitrophenol	09/11/07	41	WG-37191-082107-RN-001	53 UJ	ug/L
				WG-37191-082107-RN-002	52 UJ	ug/L
				WG-37191-082107-RN-007	50 UJ	ug/L
SVOCs	Hexachlorocyclopentadiene	09/11/07	38	WG-37191-082107-RN-001	11 UJ	ug/L
				WG-37191-082107-RN-002	10 UJ	ug/L
				WG-37191-082107-RN-007	9.9 UJ	ug/L
SVOCs	2,4-Dinitrophenol	08/03/07	34	SO-37191-072507-RN-SB-05	2000 UJ	ug/Kg
				SO-37191-072507-RN-SB-1	2300 UJ	ug/Kg
				SO-37191-072507-RN-SB-10	2000 UJ	ug/Kg
				SO-37191-072507-RN-SB-7	5300 UJ	ug/Kg
				SO-37191-072507-RN-SB-8	2100 UJ	ug/Kg
				SO-37191-072507-RN-SB-9	2000 UJ	ug/Kg
				SO-37191-072607-RN-SB-11	2200 UJ	ug/Kg
				SO-37191-072607-RN-SB-12	2100 UJ	ug/Kg
				SO-37191-072607-RN-SB-13	2000 UJ	ug/Kg
				SO-37191-072607-RN-SB-14	1900 UJ	ug/Kg
				SO-37191-072607-RN-SB-15	2000 UJ	ug/Kg
				SO-37191-072607-RN-SB-16	2200 UJ	ug/Kg
				SO-37191-072707-RN-SB-17	2400 UJ	ug/Kg
				SO-37191-072707-RN-SB-27	1900 UJ	ug/Kg
				SO-37191-072707-RN-SB-20	4400 UJ	ug/Kg
				SO-37191-072707-RN-SB-2	4100 UJ	ug/Kg
				SO-37191-072707-RN-SB-3	2000 UJ	ug/Kg
				SO-37191-072707-RN-SB-4	2100 UJ	ug/Kg
				SO-37191-073007-CB-SB6	1900 UJ	ug/Kg

Notes:
%RSD Percent Relative Standard Deviation.
SVOCs Semi-Volatile Organic Compounds.
UJ Not detected, estimated reporting limit.

PROPRIETARY DOCUMENT

TABLE 5

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING CONTINUING CALIBRATION RESULTS
GROUNDWATER AND SOIL SAMPLING
SITE
JULY - AUGUST 2007

Parameter	*Calibration Date*	*Compound*	*%D*	*Associated Sample ID*	*Qualified Sample Results*	*Units*
VOCs	08/27/07	Acetone	41	WG-37191-082107-RN-001	600 UJ	ug/L
				WG-37191-082107-RN-004	60000 UJ	ug/L
				WG-37191-082107-RN-006	20 UJ	ug/L
				WG-37191-082107-RN-007	20 UJ	ug/L
VOCs	08/28/07	Chloroethane	74	WG-37191-082107-RN-002	100 UJ	ug/L
VOCs	08/30/07	Chloroethane	84	WG-37191-082107-RN-005	1200 UJ	ug/L
VOCs	09/02/07	Chloroethane	39	WG-37191-082107-RN-003	12000 UJ	ug/L
VOCs	08/27/07	Dichlorodifluoromethane (CFC-12)	39	WG-37191-082107-RN-001	150 UJ	ug/L
				WG-37191-082107-RN-004	15000 UJ	ug/L
				WG-37191-082107-RN-006	5.0 UJ	ug/L
				WG-37191-082107-RN-007	5.0 UJ	ug/L
VOCs	09/02/07	Trichlorofluoromethane (CFC-11)	49	WG-37191-082107-RN-003	12000 UJ	ug/L
				SO-37191-072707-RN-SB-3	0.98 J	ug/Kg

Notes:

%D	Percent Difference.
J	Estimated.
SVOCs	Semi-Volatile Organic Compounds.
UJ	Not detected, estimated reporting limit.
VOCs	Volatile Organic Compounds.

TABLE 6

QUALIFIED SAMPLE RESULTS DUE TO ANALYTE CONCENTRATIONS IN THE METHOD BLANKS
GROUNDWATER AND SOIL SAMPLING
SITE
JULY - AUGUST 2007

Parameter	*Analysis Date*	*Analyte*	*Blank Result*	*Sample ID*	*Qualified Sample Result*
VOCs	08/01/07	Methylene chloride	2.1	SO-37191-072507-RN-SB-05	6.0 U
				SO-37191-072507-RN-SB-1	6.9 U
				SO-37191-072507-RN-SB-10	5.9 U
				SO-37191-072507-RN-SB-9	6.1 U
				SO-37191-072607-RN-SB-11	6.7 U
				SO-37191-072607-RN-SB-12	6.2 U
				SO-37191-072607-RN-SB-15	6.1 U
				SO-37191-072707-RN-SB-17	7.2 U
				SO-37191-072707-RN-SB-4	6.2 U
				SO-37191-072707-RN-SB-20	6.7 U
				SO-37191-072707-RN-SB-27	5.7 U
VOCs	08/01/07	Methylene chloride	56	SO-37191-072507-RN-SB-7	400 U
				SO-37191-072607-RN-SB-13	310 U
VOCs	08/01/07	Methylene chloride	1.2	SO-37191-072507-RN-SB-8	6.1 U
				SO-37191-072607-RN-SB-14	5.7 U
				SO-37191-072607-RN-SB-16	6.7 U
				SO-37191-072707-RN-SB-3	6.0 U
VOCs	08/02/07	Methylene chloride	1.5	SO-37191-073007-CB-SB18	5.2 U
VOCs	08/03/07	Methylene chloride	100	SO-37191-073007-CB-SB6	380 U
				SO-37191-073007-CB-SB19	280 U
Metals	9/7/07-ICB	Beryllium	1.0	WG-37191-082107-RN-001	4.0 U
				WG-37191-082107-RN-002	4.0 U
				WG-37191-082107-RN-007	4.0 U
Metals	08/30/07	Aluminum (Dissolved)	18.4	WG-37191-082107-RN-001	200 U
				WG-37191-082107-RN-002	200 U
				WG-37191-082107-RN-007	200 U
Metals	08/30/07	Beryllium (Dissolved)	0.65	WG-37191-082107-RN-001	4.0 U
				WG-37191-082107-RN-002	4.0 U
				WG-37191-082107-RN-007	4.0 U
Metals	8/30/07	Cadmium	0.33	WG-37191-082107-RN-001	5.0 U
				WG-37191-082107-RN-002	5.0 U

Notes:
U Not detected.
VOCs Volatile Organic Compounds.

PROPRIETARY DOCUMENT

TABLE 7

QUALIFIED SAMPLE DATA DUE TO OUTLYING SURROGATE RECOVERIES
GROUNDWATER AND SOIL SAMPLING
SITE
JULY - AUGUST 2007

Parameter	*Surrogate*	*Surrogate Recovery (percent)*	*Control Limits (percent)*	*Sample ID*	*Analytes*	*Qualified Sample Results*	*Units*
SVOCs	2-Fluorobiphenyl	99	34-97	WG-37191-082107-RN-007	bis(2-Ethylhexyl)phthalate	2.4 J	ug/L
	Nitrobenzene-d5	111	38-97		2-Methylnaphthalene	0.62 J	ug/L

Notes:
J Estimated.
SVOCs Semi-Volatile Organic Compounds.

PROPRIETARY DOCUMENT

TABLE 8

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERIES
GROUNDWATER AND SOIL SAMPLING
SITE
JULY - AUGUST 2007

Parameter	*Analyte*	*Associated Sample ID*	*MS Recovery (percent)*	*MSD Recovery (percent)*	*RPD*	*Control Limits*		*Qualified Sample Result*	*Units*
						Recovery (percent)	*RPD (percent)*		
Metals	Aluminum	WG-37191-082107-RN-001	183	168	9	75-125	30	5110 J	ug/L
		WG-37191-082107-RN-002						4190 J	ug/L
		WG-37191-082107-RN-007						8250 J	ug/L
Pesticides	4,4'-DDT	SO-37191-072507-RN-SB-1	MI	MI	-	70-130	20	1.7 J	ug/kg
General Chemistry	Cyanide (total)	SO-37191-072507-RN-SB-1	131	130	1	75-125	20	0.29 J	mg/kg
		SO-37191-072507-RN-SB-8						0.23 J	mg/kg
VOCs	Trichloroethene	SO-37191-072707-RN-SB-17	28	127	128	46-141	20	140 J	ug/kg

Notes:
- Not applicable.
J Estimated.
MI Matrix interference. Recoveries were not calculated.
MS Matrix Spike.
MSD Matrix Spike Duplicate.
RPD Relative Percent Difference.
VOCs Volatile Organic Compounds.

PROPRIETARY DOCUMENT

TABLE 9

QUALIFIED SAMPLE RESULTS DUE TO VARIABILITY IN FIELD DUPLICATE RESULTS
GROUNDWATER AND SOIL SAMPLING
SITE
JULY - AUGUST 2007

Parameter	*Analyte*	*Original Sample ID*	*Original Result*	*Duplicate Sample ID*	*Duplicate Result*	*RPD*	*Units*	*Qualifier* [1]
VOCs	cis-1,2-Dichloroethene	WG-37191-082107-RN-001	2200	WG-37191-082107-RN-002	1100	67	ug/L	J
VOCs	Trichloroethene	WG-37191-082107-RN-001	2000	WG-37191-082107-RN-002	1000	67	ug/L	J
VOCs	cis-1,2-Dichloroethene	WG-37191-082107-RN-004	65000	WG-37191-082107-RN-005	25000	89	ug/L	J
VOCs	Trichloroethene	WG-37191-082107-RN-004	190000	WG-37191-082107-RN-005	26000	152	ug/L	J
Metals	Lead	WG-37191-082107-RN-001	56.8	WG-37191-082107-RN-002	30.0	52	ug/L	J
Metals	Lead	SO-37191-072707-RN-SB-17	155	SO-37191-072707-RN-SB-27	13.4	200	mg/Kg	J

Notes:
(1) Qualifier is associated with both original and duplicate result.
J Estimated.
RPD Relative Percent Difference.
VOCs Volatile Organic Compounds.

TABLE 10

QUALIFIED SAMPLE RESULTS DUE TO DIFFERENCES IN DUAL COLUMN RESULTS
GROUNDWATER AND SOIL SAMPLING
SITE
JULY - AUGUST 2007

Parameter	*Compound*	*Associated Sample ID*	*%D*	*Reported Results*	*Units*
Pesticides	delta-BHC	WG-37191-082107-RN-007	131	0.081 U	ug/L
	Endrin aldehyde	WG-37191-082107-RN-007	82	0.051 U	ug/L

Notes:
%D Percent Difference.
BHC Benzene Hexachloride.
U Not detected.

APPENDIX B-3

REDUCED DATA VALIDATION REPORT TEMPLATE

CONESTOGA-ROVERS
& ASSOCIATES

2055 Niagara Falls Blvd., Suite #3
Niagara Falls, New York 14304
Telephone: (716) 297-6150 Fax: (716) 297-2265
www.CRAworld.com
PROPRIETARY DOCUMENT

MEMORANDUM

TO:

REF. NO.:

FROM:

DATE:

E-Mail and Hard Copy if Requested

C.C.:

RE: **Analytical Results and QA/QC Review**

Semi-Annual Groundwater Sampling Program

INTRODUCTION

Thirteen groundwater samples including one field duplicate were collected in support of the Semi-Annual Sampling Monitoring Program at the _____ Site during _____. Samples were submitted to ____ Labs, located in _____. A sample key is presented in Table 1 and the analytical parameter list and methodologies are presented in Table 2. The analytical results are summarized in Table 3. Copies of the Chain of Custody are attached.

The final results and supporting quality assurance/quality control (QA/QC) data were reviewed. Evaluation of the data was based on information obtained from the Chain of Custody forms, finished report forms, blank data, and recovery data for matrix and surrogate spikes. The QA/QC criteria by which the data have been assessed are outlined in the respective methods and the following documents:

i) "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October 1999, United States Environmental Protection Agency (USEPA) 540/R-99/008; and

ii) "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," February 1994, USEPA 540/R-94/013.

QA/QC REVIEW

Sample holding times were assessed against the criteria outlined in Table 2. All holding time criteria were met, and all samples were properly preserved and maintained at 4°C (±2°C).

Method blanks were analyzed with the investigative samples for all parameters. All method blank results were non-detect for the compounds of interest.

All samples and blanks were spiked with surrogate compounds prior to sample extraction and/or analysis in accordance with the organic methods. All surrogate spike recoveries met the associated method criteria

indicating adequate analytical efficiency with the exception of a high recovery for diesel range organics. All associated positive sample results were qualified as estimated based on the implied high bias (see Table 4).

A blank spike and/or blank spike duplicate (BS and/or BSD) was analyzed for all parameters. All recoveries were within the laboratory control limits indicating good analytical accuracy and/or precision.

A matrix spike/matrix spike duplicate (MS/MSD) was analyzed for dissolved iron, ferrous iron, nitrate, sulfate, and total alkalinity. All recoveries were within the laboratory control limits indicating good analytical accuracy and precision, with the exception of low recoveries for sulfate. All associated sample results were qualified as estimated based on the implied low bias (see Table 5).

Trip blanks were collected and analyzed with the investigative samples for gasoline range organics. All trip blank results were non-detect for the compounds of interest.

A field duplicate sample was collected and analyzed as shown in Table 1. All results showed good precision.

CONCLUSION

Based on this QA/QC review, the data summarized in Table 3 were judged to be acceptable with the qualifications noted.

PROPRIETARY DOCUMENT

TABLE 1

SAMPLING AND ANALYSIS SUMMARY
SEMI-ANNUAL GROUNDWATER SAMPLING PROGRAM

				Analysis/Parameters								
Sample ID	*Location ID*	*Collection Date (mm/dd/yy)*	*Collection Time (hr:min)*	*TPHg (BTEX, Gasoline)*	*TPHd (Motor Oil, Diesel)*	*Dissolved Iron*	*Ferrous Iron*	*Total Alkalinity*	*Nitrate*	*Sulfate*	*Carbon Dioxide, Methane*	*Comments*
GW020807JSMW110	MW-110	02/08/07	11:00:00	X	X	X	X	X	X	X	X	
GW020807JSMW106	MW-106	02/08/07	12:50:00	X	X	X	X	X	X	X	X	
GW020807JSMW109	MW-109	02/08/07	15:40:00	X	X	X	X	X	X	X	X	
GW020807JSMW108	MW-108	02/08/07	17:10:00	X	X	X	X	X	X	X	X	
GW021107JSMW107	MW-107	02/12/07	9:55:00	X	X	X	X	X	X	X	X	
GW021107JSMW105	MW-105	02/12/07	10:40:00	X	X	X	X	X	X	X	X	
GW021107JSMW103	MW-103	02/12/07	13:22:00	X	X	X	X	X	X	X	X	
GW021107JSMW102	MW-102	02/12/07	14:59:00	X	X	X	X	X	X	X	X	
GW021307JSMW104	MW-104	02/13/07	12:23:00	X	X	X	X	X	X	X	X	
GW021307JSMW904	MW-104	02/13/07	14:32:00	X	X	X	X	X	X	X	X	Field duplicate of GW021307JSMW104
GW021307JSMW104-26	MW-104 @ 26 feet	02/13/07	10:23:00	X								
GW021307JSMW104-30	MW-104 @ 30 feet	02/13/07	10:27:00	X								
GW021307JSMW104-34	MW-104 @ 34 feet	02/13/07	10:39:00	X								

Notes:
BTEX Benzene, Toluene, Ethylbenzene, and Xylene.
TPH Total Petroleum Hydrocarbons.

TABLE 2

ANALYTE PARAMETER LIST
SEMI-ANNUAL GROUNDWATER SAMPLING PROGRAM

Analytical Parameter	*Method Number*
DRO - Diesel/Motor Oil	USEPA 8015B/3501C [1]
GRO /BTEX	USEPA 8015B/5030B [1]
Dissolved Iron	USEPA 6020 [1]
Ferrous Iron	SM 3500 Fe D[2]
Alkalinity (as CaCO3)	SM 2320B[2]
Nitrate	USEPA 353.2 [3]
Sulfate	USEPA 300.0[3]
Carbon Dioxide	RSK-175[4]
Methane	RSK-175[4]

Notes:

(1) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," 3rd Edition, November, 1986 (with all subsequent revisions).

(2) 18th Edition, 1992 (with subsequent revisions).

(3) "Methods for Chemical Analysis of Water and Wastes," USEPA 600/4-79-(

(4) USEPA Internal Standard Operating Procedure dated August 11, 1994 by Bryan Newell at the R. S. Kerr Laboratory in Oklahoma.

BTEX Benzene, Toluene, Ethylbenzene, and Xylene.

DRO Diesel Range Organics.

GRO Gasoline Range Organics.

USEPA United States Environmental Protection Agency.

PROPRIETARY DOCUMENT

TABLE 3

ANALYTICAL RESULTS SUMMARY
SEMI-ANNUAL GROUNDWATER SAMPLING PROGRAM

Sample Location:		*MW-102*	*MW-103*	*MW-104*	*MW-104*	*MW-104*	*MW-104*	*MW-104*
Sample ID:		*GW021207JSMW102*	*GW021207JSMW103*	*GW021307JSMW104*	*GW021307JSMW904*	*GW021307JSMW104-26*	*GW021307JSMW104-30*	*GW021307JSMW104-34*
Sample Date:		*2/12/2007*	*2/12/2007*	*2/13/2007*	*2/13/2007*	*2/13/2007*	*2/13/2007*	*2/13/2007*
					Duplicate			
Parameters	*Units*							
Petroleum Products								
Benzene	µg/L	36	0.5 U	530	510	53	1400	460
Ethylbenzene	µg/L	0.5 U	0.5 U	11	11	0.5 U	5.9	8.2
Toluene	µg/L	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
Xylene (total)	µg/L	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
Diesel Fuel	µg/L	330	330	390 J	410 J	-	-	-
Gasoline	µg/L	140	20 U	1200	1200	130	3100	1000
TPH-Motor Oil	µg/L	250 U	310	250 U	250 U	-	-	-
Metals								
Iron (Dissolved)	µg/L	820	236	6410	6520	-	-	-
Ferrous Iron	mg/L	1.0	1.0 U	10.7	10.8	-	-	-
Gas								
Carbon dioxide	µg/mL	200	120	240	240	-	-	-
Methane	µg/mL	0.36	0.036	0.02	0.021	-	-	-
General Chemistry								
Alkalinity, Total (as CaCO3)	mg/L	998	398	1400	1400	-	-	-
Nitrate (as N)	mg/L	0.43	0.45	0.60	0.52	-	-	-
Sulfate	mg/L	7590 J	1270 J	10600 J	10400 J	-	-	-

Sample Location:		*MW-105*	*MW-106*	*MW-107*	*MW-108*	*MW-109*	*MW-110*
Sample ID:		*GW021207JSMW105*	*GW020807JSMW106*	*GW021207JSMW107*	*GW020807JSMW108*	*GW020807JSMW109*	*GW020807JSMW110*
Sample Date:		*2/12/2007*	*2/8/2007*	*2/12/2007*	*2/8/2007*	*2/8/2007*	*2/8/2007*
Parameters	*Units*						
Petroleum Products							
Benzene	µg/L	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
Ethylbenzene	µg/L	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
Toluene	µg/L	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
Xylene (total)	µg/L	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
Diesel Fuel	µg/L	50 U	590	480	50 U	50 U	50 U
Gasoline	µg/L	20 U	20 U	20 U	20 U	20 U	20 U
TPH-Motor Oil	µg/L	250 U	250 U	250 U	250 U	250 U	250 U
Metals							
Iron (Dissolved)	µg/L	4260	50 U	228	50 U	132	1570
Ferrous Iron	mg/L	7.7	1.0 U	1.0 U	1.0 U	1.0 U	0.89J
Gas							
Carbon dioxide	µg/mL	67.0	59.0	60.0	58.0	16.0	110
Methane	µg/mL	0.010 U	0.010 U	0.010 U	0.010 U	0.34	0.010 U
General Chemistry							
Alkalinity, Total (as CaCO3)	mg/L	388	179	386	345	464	815
Nitrate (as N)	mg/L	2.0	3.9	1.4	4.5	3.5	2.3
Sulfate	mg/L	15900 J	24.5 J	2390 J	532 J	2310 J	1240 J

Notes:
- Not analyzed.
J Estimated.
TPH Total Petroleum Hydrocarbon.
U Non-detect at associated value.

TABLE 4

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING SURROGATE RECOVERIES
SEMI-ANNUAL GROUNDWATER SAMPLING PROGRAM

Parameter	*Surrogate*	*Surrogate Recovery (percent)*	*Control Limits (percent)*	*Sample ID*	*Analyte*	*Qualified Sample Results*	*Units*
TPH	Octacosane	145	28-142	GW021307JSMW104	Diesel Fuel	390 J	µg/L
				GW021307JSMW904		410 J	µg/L

Notes:
J Estimated.
TPH Total Petroleum Hydrocarbons.

PROPRIETARY DOCUMENT

TABLE 5

QUALIFIED SAMPLE RESULTS DUE TO OUTLYING MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERIES
SEMI-ANNUAL GROUNDWATER SAMPLING PROGRAM

Parameter	*Associated Sample ID*	*Analyte*	*MS Recovery (percent)*	*MSD Recovery (percent)*	*RPD*	*Control Limits*		*Qualified Sample Result*	*Units*
						Recovery (percent)	*RPD (percent)*		
General Chemistry	GW020807JSMW110	Sulfate	75	74	0.3	90-110	20	1240 J	mg/L
	GW020807JSMW106							24.5 J	mg/L
	GW020807JSMW108							532 J	mg/L
	GW020807JSMW109							2310 J	mg/L
General Chemistry	GW021207JSMW107	Sulfate	60	60	0.0	90-110	20	2390 J	mg/L
	GW021207JSMW102							7590 J	mg/L
	GW021207JSMW103							1270 J	mg/L
	GW021207JSMW105							15900 J	mg/L
General Chemistry	GW021307JSMW104	Sulfate	67	68	0.43	90-110	20	10600 J	mg/L
	GW021307JSMW904							10400 J	mg/L

Notes:
J Estimated.
MS Matrix Spike.
MSD Matrix Spike Duplicate.
RPD Relative Percent Difference.

APPENDIX K-K

SHALLOW GROUNDWATER WORK PLAN

CONESTOGA-ROVERS
& ASSOCIATES

651 Colby Drive, Waterloo, Ontario, Canada N2V 1C2
Telephone: (519) 884-0510 Facsimile: (519) 884-0525
www.CRAworld.com

December 17, 2010 Reference No. 038443-89

Ms. Karen Cibulskis
Remedial Project Manager
United States Environmental Protection Agency
Region V
77 West Jackson Boulevard
Mail Code SR-6J
Chicago, IL 60604

Dear Ms. Cibulskis:

Re: Shallow Groundwater Work Plan (Work Plan)
South Dayton Dump and Landfill Site Moraine, Ohio (Site)

As required under the Dispute Resolution Agreement signed by the Respondents and USEPA on December 10, 2010, this Work Plan presents the proposed approach for additional investigation of shallow groundwater conditions at the Site boundary between VAS-09 and VAS-22 and in the vicinity of MW-210 (Shallow Groundwater Investigation). Conestoga-Rovers & Associates (CRA) has prepared this Work Plan on behalf of the Respondents to the Administrative Settlement Agreement and Order on Consent (ASAOC) for Remedial Investigation/Feasibility Study (RI/FS) of the Site, Docket No. V-W-06-C-852 (Respondents).

The work proposed in this Work Plan will be performed in accordance with the United States Environmental Protection Agency- (USEPA-) approved Field Sampling Plan (FSP), Quality Assurance Project Plan (QAPP), and Site-Specific Health and Safety Plan (HASP), and associated addenda that are submitted as attachments to this Work Plan.

This Work Plan is presented in the following titled sections:

1.0 Background
2.0 Shallow Groundwater Investigation
3.0 Schedule
4.0 Reporting

1.0 BACKGROUND

The Respondents to the ASAOC include Hobart Corporation (Hobart), Kelsey Hayes Company (Kelsey-Hayes), and NCR Corporation (NCR). These three Respondents (the PRP Group) are and have been performing the Work required by the ASAOC under the direction and oversight of the USEPA.

Under the December 10, 2010 Dispute Resolution Agreement, the Respondents agreed to investigate the shallow groundwater along the Site boundary between VAS-09 and VAS-22 and in the vicinity of monitoring well MW-210. This investigation is to identify potential risks to off-Site receptors from volatile organic compounds (VOCs) and naphthalene migrating off-Site in groundwater and into buildings via the vapor intrusion pathway.

Specifically, the Dispute Resolution Agreement requires the Respondents to:

> *submit a work plan (Shallow Groundwater Work Plan) including FSP and QAPP Addenda, for additional characterization of the top five feet of shallow groundwater in the vicinity of Monitoring Well 210 (MW-210) at the locations in the Respondents' draft MW-210 Shallow Groundwater Investigation Letter Work Plan, dated March 16, 2010, and at locations no greater than 100 feet apart at the Site boundary starting: 1. adjacent to Dryden Road east of VAS-09; 2. continuing south to the Site boundary at the intersection of Dryden Road and East River Road; 3. continuing west along the south side of the access road to Lot 4610, with a sampling point at the northeast corner of Lot 4610; 4. continuing south along the east boundary of Lot 4610 to Lot 3254 (skipping the Site boundary around Lot 3252); and 5. continuing southwest along the East River Road boundary of the Site to a location east of VAS-22 (Shallow Groundwater Investigation Letter Work Plan). See highlighted area on [Figures 1 and 2], attached, for an illustration of the sampling area. The data quality objectives for the groundwater samples will include, but are not limited to, detecting VOCs and naphthalene in shallow groundwater at the Site boundary that pose more than a 1×10^{-6} cancer risk or a hazard index greater than 1.0 through the vapor intrusion pathway to current or potential future receptors. The samples may be collected using direct push technology, and will be collected using low-flow sampling and groundwater stabilization procedures consistent with those developed for the vertical aquifer sampling previously conducted during RI/FS Work at the Site provided the low-flow sampling and groundwater stabilization procedures meet the data quality objectives required for the VI Study. The sampling intake will be set approximately 2.5 feet below the water table. This Shallow Groundwater Work Plan for additional characterization of groundwater shall be submitted by December 17, 2010.*

The PRP Group prepared this Letter Work Plan based on requirements of the Dispute Resolution Agreement, previous investigation results and discussions between the PRP Group and USEPA.

2.0 SHALLOW GROUNDWATER INVESTIGATION

The general objective of the Shallow Groundwater Investigation is to identify whether contaminants are present in the upper five feet of shallow groundwater at the Site boundary between VAS-09 and VAS-22 that pose more than a 1×10^{-6} cancer risk or a hazard index (HI) greater than 1 to current or potential future receptors via the vapor intrusion pathway. This will be accomplished by collecting and analyzing groundwater samples from new borings completed in select locations between VAS-09 and VAS-22, as shown on Figure 1.

The Shallow Groundwater Investigation will also include the collection of a groundwater sample from the water supply well located 500 feet downgradient from MW-210. The groundwater sample from the water supply well will be analyzed for VOCs, naphthalene, and metals.

Shallow Groundwater Investigation Data Quality Objectives

There are seven steps in the Data Quality Objective (DQO) process[1]. A discussion of the DQO steps for the Shallow Groundwater Investigation is presented below.

Step 1: State the Problem – VOCs and naphthalene are present in shallow groundwater beneath the Site. A data gap exists with respect to whether VOCs and naphthalene contaminants in OU1 shallow groundwater between VAS-09 and VAS-22 are migrating off-Site in this area at concentrations that may pose an unacceptable risk to current or potential future receptors via the vapor intrusion pathway.

Step 2: Identify the goals of the study – Complete a screening level investigation to determine whether contaminants identified in the specified areas are migrating off-Site via shallow groundwater at concentrations that may pose an unacceptable risk to current or potential receptors via the vapor intrusion pathway. Identify areas where off-Site migration is occurring and further investigation, e.g., installation of additional boreholes, permanent monitoring wells or soil gas probes, or remedial activities, is required.

[1] As detailed in the USEPA document *Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA QA/G-4, February 2006.*

Step 3: Identify information inputs – Complete groundwater investigations using direct push technology and low flow groundwater sampling to determine VOC and naphthalene concentrations in shallow groundwater at discrete locations along the Site boundary.

Step 4: Identify the boundaries of the study – The Study Area for the shallow groundwater investigation is detailed below, and presented on Figure 1.

- In the vicinity of monitoring well MW-210
- At locations no greater than 100 feet (ft) apart at the Site boundary starting
  - Adjacent to Dryden Road, east of VAS-09
  - Continuing south to the Site boundary at the intersection of Dryden Road and East River Road
  - Continuing west along the south side of the access road to Lot 4610, with a sampling point at the northeast corner of Lot 4610
  - Continuing south along the east boundary of Lot 4610 to Lot 3254 (skipping the Site boundary around Lot 3252)
  - Continuing southwest along the East River Road boundary of the Site to a location east of VAS-22

Step 5: Develop the analytic approach – Groundwater samples will be collected using low flow sampling techniques from the top five feet of shallow groundwater in each borehole, following purging and stabilization. The sample intake will be set at 2.5 feet below the water table. Samples will be collected using the sampling methodologies outlined in the FSP and relevant addenda. Groundwater samples will be submitted for analysis of VOCs and naphthalene using the analytical methodologies outlined in the QAPP.

Step 6: Specify Performance or Acceptance Criteria – Performance criteria consist of identifying VOCs and naphthalene that pose more than a 1×10^{-6} cancer risk or a HI greater than 1 to current or potential future receptors via the vapor intrusion pathway. The maximum width of a groundwater plume containing VOCs at concentrations greater than MCLs at the Site boundary that may escape detection during the investigation is 100 feet over most of the Site boundary between VAS-09 and VAS-22 and is 20 feet in the vicinity of MW-210 (see Figure 1).

Step 7: Develop the plan for obtaining data – See Sections 2.1 to 2.3 below, for detailed procedures proposed in order to obtain the required data.

Vapor Intrusion (VI) is the migration of volatile chemicals from the subsurface into overlying buildings. VI is a potential concern at any building, existing or planned, located near soil or

groundwater contaminated with toxic chemicals that can volatilize. USEPA's 2002 draft guidance document, entitled "OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (OSWER Draft Guidance), defines "near" as:

> *volatile or toxic compounds within 100 ft (laterally or vertically) of buildings, unless there is a conduit that intersects the migration route that would allow soil gas to migrate further than 100 ft.*
>
> The OSWER Draft Guidance defines a conduit as: "*any passageway that could facilitate flow of soil gas, including porous layers such as sand or gravel, buried utility lines, and animal burrows.*"

At the commencement of the Shallow Groundwater Investigation, CRA will visually assess the Site boundary within the Study Area for evidence of conduits that might facilitate the flow of soil gas and will install and sample the groundwater from boreholes located immediately adjacent to any such conduit, in addition to the boreholes discussed above and shown on Figures 1 and 2.

2.1 Chlorinated Solvent Delineation for Shallow Groundwater in the Area of MW-210

The highest TCE concentrations in groundwater from any permanent monitoring well are the samples collected from MW-210. Groundwater impacts at VAS-21 and monitoring wells MW-210, MW-210A, and MW-210B are well defined vertically to 200 feet below ground surface[2]. However, the source and extent of impact present in the shallow groundwater at MW-210 are not well understood and it is not known if contaminants present in shallow groundwater at MW-210 are migrating off-Site. As such, the Respondents propose to complete additional investigation to delineate groundwater impact in this area.

CRA will advance seven boreholes on Site to the south and east of the MW-210 monitoring well nest at an initial distance interval of 20 feet along the southern fence line. To the north of the MW-210 monitoring well nest, CRA will advance four boreholes at an initial distance interval of 40 feet. Boreholes will be advanced to the top five feet of shallow groundwater, to a maximum depth of 40 feet (i.e., to the top of the till layer[3]). Figure 2 presents the approximate locations of the proposed boreholes around MW-210.

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[2] CRA notes that the full vertical extent of the deeper vinyl chloride contamination (i.e., beyond 200 feet below ground surface) has not been fully delineated. Delineation of the vertical extent of deep groundwater contamination will be completed during the OU2 RI.

[3] If the top of the till layer is located at a depth greater than 40 ft, CRA will attempt to advance the boreholes deeper than 40 ft if groundwater is not encountered within the first 40 ft.

2.2 Shallow Boundary Groundwater Investigation

The VOC concentrations in shallow groundwater in the vicinity of VAS-09 may be linked to the VOC concentrations in groundwater samples collected from MW-215A and VAS-15, and concentrations in soil gas samples collected from GP12-09, GP13-09, and GP14-09. The source of the VOC concentrations in shallow groundwater in the vicinity of VAS-09 has not been identified.

CRA collected a soil gas sample from GP09-09 that contained TCE at a concentration of 2,000 $\mu g/m^3$. This concentration indicates that there may be a source of TCE on Lot 4610. GP09-09 is located approximately 150 feet from a residential property, and approximately 200 feet from a house with a basement foundation. GP09-09 is located at the Site boundary on Lot 4610, the location of the former Mantle Oil Service facility. Between 1971 and 1986 Mantle Oil Service operated a used oil reclamation service on Lot 4610 of the Site including 18 above ground storage tanks.

In order to determine the concentration of shallow groundwater contaminants at the Site boundary within the Study Area, CRA will advance boreholes at distances no greater than 100 feet as shown on Figure 1. CRA will collect a groundwater sample from the top five feet of shallow groundwater at each borehole following the procedures detailed in Section 2.3.

2.3 Borehole Advancement

The proposed groundwater sampling locations are shown on Figures 1 and 2. All borings will be completed using Geoprobe™ direct push drilling techniques. Details regarding Geoprobe™ drilling are provided in Attachment A (addendum to the FSP).

The drill rods, stainless steel screen, and associated drilling equipment will be decontaminated, prior to starting and between each borehole, using a high-pressure, high temperature, hot water cleaner. An off-Site source of potable water, free of contamination such as a fire hydrant will be used. CRA previously collected a sample of the potable water source for analysis of VOCs to verify water quality. In the event of a change in the potable water source, CRA will collect a sample from the new source.

During borehole advancement, continuous soil cores will be retrieved to log soil stratigraphy. Cores will be screened with a photoionization detector (PID) for the presence of volatile organic compounds (VOCs), and screened for the presence of methane, either by using a landfill gas meter (such as a Landtec GEM-500 or MultiRAE 4-Gas monitor) or a flame-ionization detector (FID) calibrated for methane.

Where field screening indicates evidence of contamination, soils will be tested for the presence of NAPL using a Sudan IV® dye test. Field calibration, preventative maintenance, and SOPs for the PID and Sudan IV® dye test are included in the FSP.

Following the field screening and logging of the soil stratigraphy at each borehole, the Geoprobe will be offset approximately 1 foot from the borehole to collect a groundwater sample while preventing drawdown. CRA proposes to use a Geoprobe Screen Point 16 (SP16) Groundwater Sampler. The standard operating procedure (SOP) for the Geoprobe SP16 Groundwater Sampler is included in Attachment A (addendum to the FSP). CRA proposes to use a 32-inch (2.6-ft) stainless steel slotted screen with the Geoprobe SP16 Groundwater Sampler. CRA will collect groundwater samples from the top five feet of shallow groundwater. The sampling intake will be set approximately 2.5 feet below the water table, with the top of the 32-inch stainless steel screen set approximately 1.25 ft below the water table in order for the sampling intake to be set at the midpoint of the screen.

Groundwater samples will be collected through the stainless steel screen using a mechanical bladder pump set at a flow rate of 100 milliliters per minute (mL/min). The SOP for the mechanical bladder pump is included in Attachment A (addendum to the FSP).

The flow rate for purging of groundwater will be dependent on the capacity of the mechanical bladder pump and the transmissivity of the aquifer material. Efforts will be made to maintain low flow during purging (i.e., 100 to 500 mL/min for purging). The minimum required water volume (i.e., three to five screen volumes) will be purged at the lowest sustainable flow rate. During the screen purging, field parameters such as pH, temperature, specific conductance, and turbidity will be monitored to evaluate the stabilization of the purged groundwater. The groundwater will be considered stable after a maximum of five well screen volumes are removed or when three successive readings for pH, specific conductance, and temperature agree within the following limits:

- pH: ±0.1 pH units
- specific conductance: ±3 percent (temperature corrected)
- temperature: ±1.0 °C

pH, and temperature will be monitored using a YSI Model 3560 instrument. Turbidity will be measured using a HF Scientific DRT-15C Turbidimeter. Alternatively, equivalent instruments may be used.

For sampling intervals where the nature of the formation substantially restricts the flow of water during purging, purging will continue for a maximum of two hours. Groundwater

samples will be collected once the parameters have stabilized as detailed in the FSP, or once the maximum purging time has been reached. Groundwater samples will not be collected if attempts to purge and sample indicate the interval does not yield enough water to sample. If this occurs, the borehole location will be resituated, and another attempt to collect a groundwater sample will be made.

All groundwater samples will be analyzed for Target Compound List (TCL) VOCs and naphthalene on a regular (five-day) turnaround time basis.

The groundwater sample from the water supply well located 500 feet downgradient from MW-210 will be analyzed for TCL VOCs, naphthalene, and metals on a regular turnaround time basis.

For QA/QC purposes, CRA will submit one field duplicate for every 10 groundwater samples submitted. Based on the total expected number of groundwater samples to be collected during borehole advancement, CRA will submit three field duplicate groundwater samples. CRA will also submit one trip blank sample per shipment, for VOC analyses to assess the sample handling procedures.

The results of the Shallow Groundwater Investigation will be evaluated to identify locations within the study boundary where concentrations of VOCs or naphthalene in shallow groundwater at the Site boundary exceed the VI pathway 1×10^{-6} cancer risk or a HI of 1 for a residential exposure scenario. Following completion of the Shallow Groundwater Investigation, CRA will recommend any additional temporary boreholes, permanent monitoring wells, soil vapor investigation, or remedial activities required in order to further define or mitigate unacceptable risks posed by contaminants in shallow groundwater at the Site boundary between VAS-09 and VAS-22. Any additional investigation that is deemed necessary based on the results of the Shallow Groundwater Investigation will be completed on an expedited basis outside of the OU2 Remedial Investigation process unless otherwise agreed between the Respondents and USEPA.

3.0 SCHEDULE

Field work will begin within three weeks of receipt of USEPA approval of the Shallow Groundwater and VI Investigation Letter Work Plan, dependant on drilling subcontractor availability, and obtaining access to the various private properties and businesses.

4.0 REPORTING

CRA will post the validated analytical results to the South Dayton Dump and Landfill file transfer protocol (ftp) site immediately upon validation. Stratigraphic information will also be posted to the ftp site as soon as it is compiled from the field notes. The draft Shallow Groundwater and VI Investigation Report will be submitted to USEPA within 30 days of receipt of the final laboratory data report.

The draft Shallow Groundwater Investigation Report will provide a summary of results from the Shallow Groundwater Investigation and recommendations for further sampling or remedial actions required to identify and address any unacceptable risks to on- or off-Site receptors. The Shallow Groundwater Investigation Report will be finalized following receipt of comments from USEPA. Monthly progress reports submitted to USEPA during the investigative work will include the information required for monthly progress reports in the RI/FS SOW.

Should you have any questions on the above, please do not hesitate to contact us.

Yours truly,

CONESTOGA-ROVERS & ASSOCIATES

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VC/ca/96
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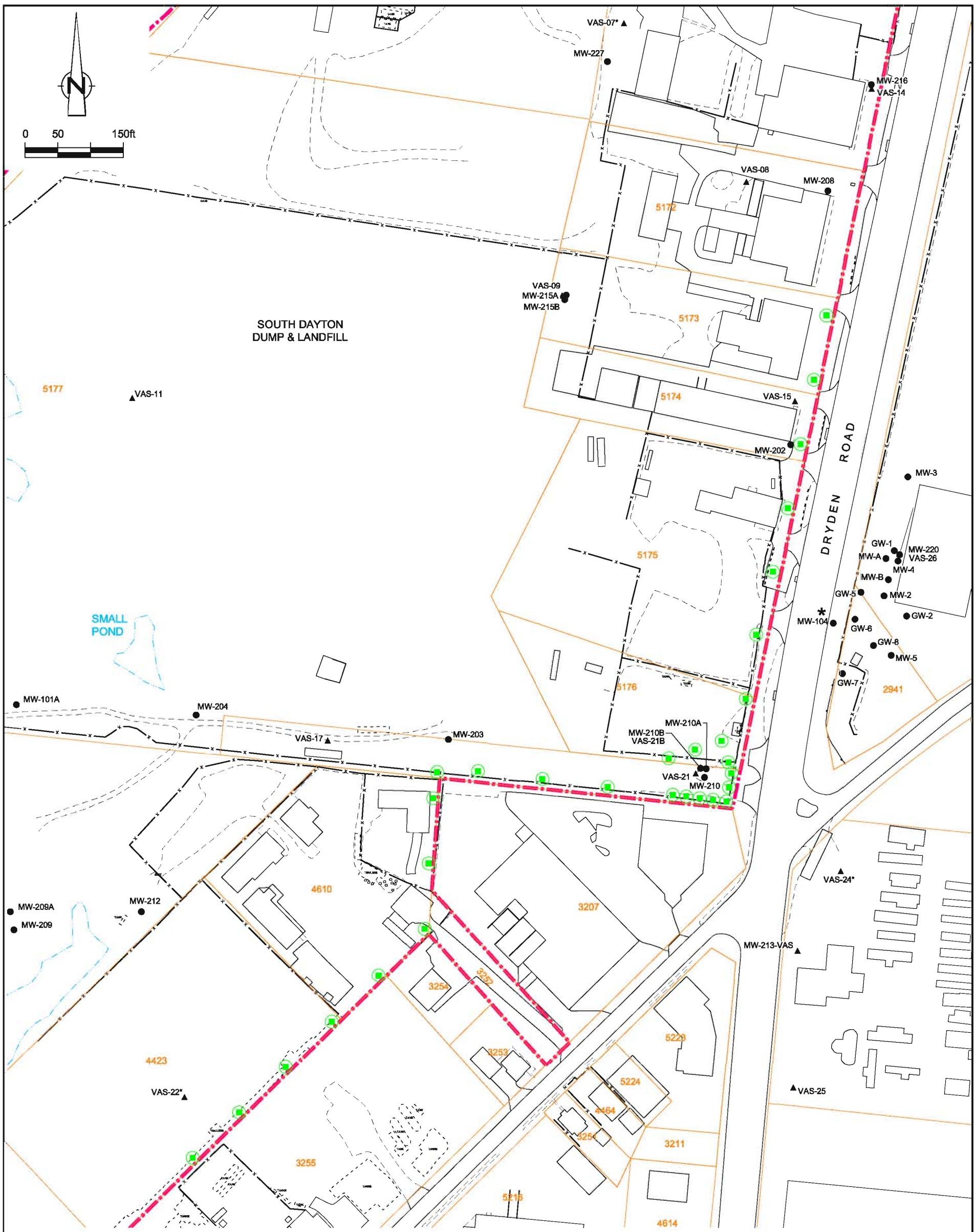


LEGEND

- APPROXIMATE SITE BOUNDARY
- EDGE OF WATER
- PARCEL BOUNDARY
- 3896 PARCEL NUMBER
- MW-206 ● UPPER AQUIFER MONITORING WELL LOCATION
- VAS-01 ▲ VERTICAL AQUIFER SAMPLING LOCATION
- • VAS LOCATION COMPLETED TO 70 FEET BELOW GROUND SURFACE OR LESS
- PROPOSED GROUNDWATER SAMPLING LOCATION

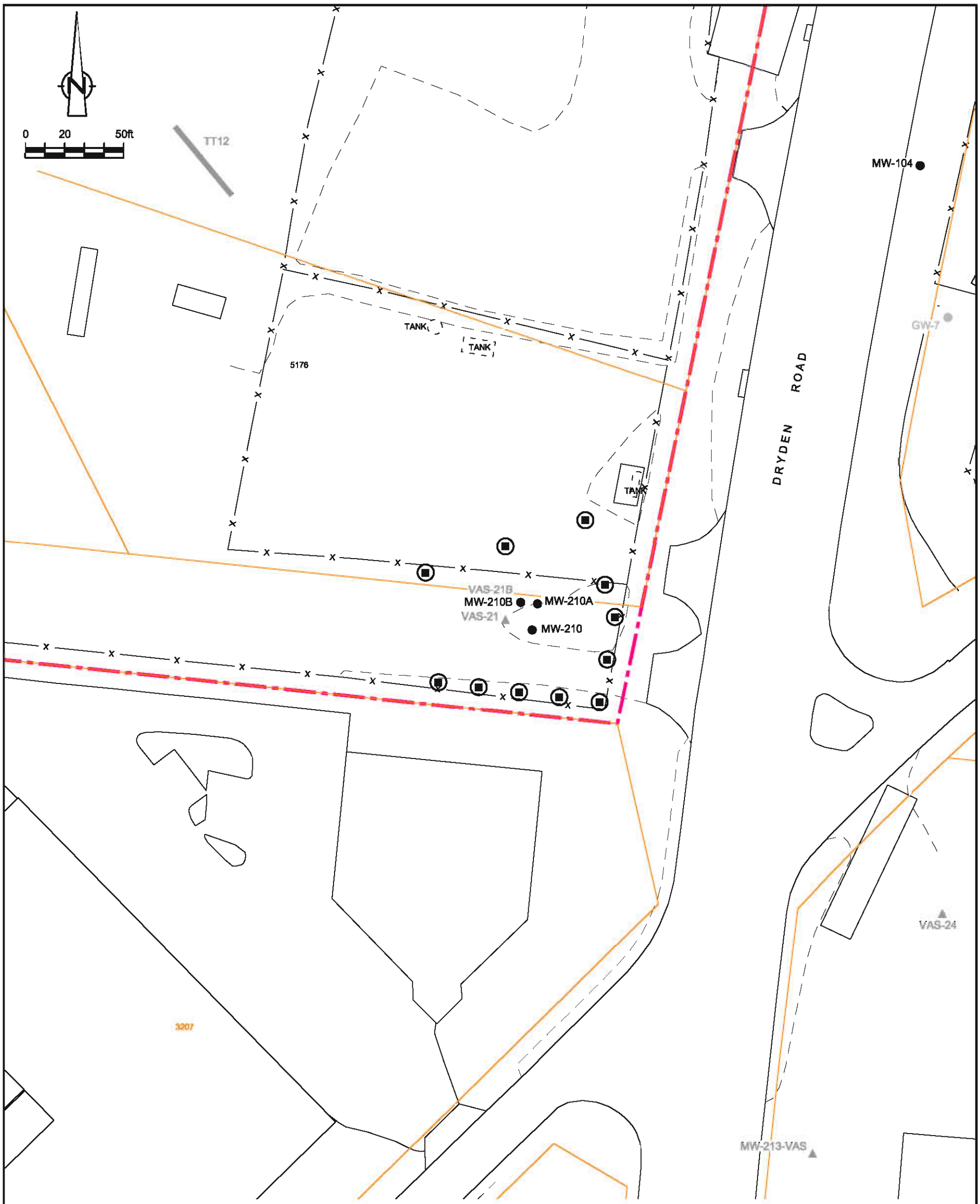
SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.
ABRAMS AERIAL SURVEY INC. PROJECT 38443, AASI 29610, 04/02/2008

figure 1

PROPOSED GROUNDWATER SAMPLING LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE

Moraine, Ohio

38443-61(CIBU096)GN-WA001 DEC 17/2010



LEGEND

- SITE BOUNDARY (SOW 2006)
- EDGE OF WATER
- PARCEL BOUNDARY
- MW-206 MONITORING WELL LOCATION
- MW-1 HISTORIC DP&L MONITORING WELL LOCATION
- TT1 TEST TRENCH LOCATION
- VAS01 VERTICAL AQUIFER SAMPLING LOCATION
- PROPOSED MEMBRANE INTERFACE PROBE (MIP) BOREHOLE LOCATION (NOTE THAT ADDITIONAL LOCATIONS MAY BE ADDED BETWEEN THE LOCATIONS SHOWN TO BETTER DELINEATE THE PLUME)

figure 2

PROPOSED MW-210 BOREHOLE LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.
ABRAMS AERIAL SURVEY INC. PROJECT 38443, AASI 29610, 04/02/2008

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ATTACHMENT A

VERTICAL AQUIFER SAMPLING/TEMPORARY MONITORING
WELL INSTALLATION AND SAMPLING BY GEOPROBE®

ATTACHMENT A

STANDARD OPERATING PROCEDURE FOR VERTICAL AQUIFER SAMPLING /TEMPORARY MONITORING WELL INSTALLATION AND SAMPLING BY GEOPROBE®

1.0 INTRODUCTION

Shallow vertical aquifer sampling (VAS) boreholes and temporary monitoring wells may be installed via direct-push Geoprobe methods.

Direct-push (a.k.a., Geoprobe) refers to the sampler being "pushed" into the soil material without the use of drilling to remove the soil. This method relies on the drill unit static weight, combined with rapid hammer percussion, to advance the tool string. Discrete soil samples are continuously obtained. It is important that the direct-push drilling method (i.e., Geoprobe) used minimizes the disturbance of subsurface materials.

This method is used extensively for initial site screening to establish site geology and delineate vertical and horizontal plume presence.

Standard Penetration Test (SPT) blow count values cannot be obtained when sampling with a direct-push discrete soil sampler.

The direct-push method is popular due to the limited volume of cuttings produced and the speed of the sampling process, which can be much faster than the sample description and sample preparation process.

Discrete continuous soil samples are collected in tube samplers (various lengths) affixed with a cutting shoe and internal liner [polyvinyl chloride (PVC), Teflon, or acetate are available]. The soil sampler may be operated in "open-mode" (when borehole collapse is not a concern), or in "closed-mode" (when minimization of sample "slough" is desired). Closed-mode operation involves placement of a temporary drill-point in the cutting shoe and driving the assembled sampler to depth. At the required depth, the temporary drill–point is released (via internal threading) and the sampler is driven to the desired soil interval. The drill-point slides inside the sample liner, riding above the collected soil column. Once driven to depth, the sampler is retrieved to the ground surface and the sample liner, with soil, is removed for examination. Extra care must be taken when cutting open the sample tube; no open blade cutting tools may be used in the process, you must have an appropriate stabilizer/holder for the tube, and cut resistant hand protection must be included as part of the overall PPE.

The Geoprobe drilling method should not contaminate the subsurface soils and groundwater. It is extremely important that drilling does not create a hydraulic link or conduit between different hydrostratigraphic units. Groundwater in monitoring and extraction wells must not be contaminated by drilling fluids or the borehole advancement process. Geoprobe drilling equipment will be decontaminated before use and between locations to prevent cross-contamination between VAS boreholes or temporary monitoring well locations and sites. Geoprobe drilling equipment will be decontaminated between well locations regardless of whether or not contaminants are suspected. Section 7.0 in the FSP specifies the required decontamination procedures. At a minimum, decontamination procedures detailed in Section 7.0 of the FSP should be used during monitoring well design and construction.

A Geoprobe SP16 will be used for shallow VAS and temporary monitoring well activities. The SOP for the Geoprobe SP16 Groundwater Sampler is included in Attachment A (addendum to the FSP). The Geoprobe SP16 is a direct push groundwater sampling device that consists of a well screen inside a steel sheath that is driven to the desired sample depth using standard Geoprobe rods. The Geoprobe SP16 is then deployed by retracting the steel sheath and exposing the well screen directly to the formation. The maximum well screen length of the Geoprobe SP16 is 32 inches. Generally, the full 32-inch well screen will be used for VAS and temporary monitoring well activities. Groundwater samples will be collected through the stainless steel screen using a mechanical bladder pump set at a flow rate of 100 milliliters per minute (mL/min) (a peristaltic pump may also be use). The SOP for the mechanical bladder pump is included in Attachment A (addendum to the FSP). The VAS or temporary monitoring well location will then be abandoned using grouting.

Finally, if a permanent monitoring well is required, pre-cleaned construction materials are used in order to prevent the potential introduction of contaminants into a hydrostratigraphic unit. Permanent monitoring well installation is discussed in Section 2.7 of the FSP.

2.0 PLANNING AND PREPARATION

Prior to undertaking shallow VAS or temporary groundwater monitoring well installation and sampling utilizing a Geoprobe the following procedures will be followed:

1. Review the appropriate Work Plan and Site-Specific Heath and Safety Plan (HASP), project documents, all available geologic and hydrogeologic mapping and reports, water well records, and historic site reports to become familiar with

the geologic and hydrogeologic framework of the site and surrounding area. Review and become familiar with the health and safety requirements, and discuss the work activities with the Project Coordinator.

2. Assemble all required equipment, materials, log books, and forms.
3. Obtain a site plan and previous stratigraphic logs. Determine the exact number, location, and depth of wells to be installed.
4. If not performed as part of borehole advancement, complete a Property Access/Utility Clearance Data Sheet. In most instances, the utility clearances and property access will have been completed as part of the well drilling and advancements.
5. Determine notification requirements with the Project Coordinator. Have all regulatory groups, the client, landowner, drilling contractor, and CRA personnel been informed of the well design and installation program?
6. Determine the methods for handling and disposal of cuttings, purged groundwater, and decontamination fluids. Generally, this is dealt with as part of the well advancement activities.

In addition to the above, the following may be required when conducting VAS or temporary monitoring well installation and sampling activities:

1. Establish a water source for well installation and decontamination. Pre-plan the methods of handling and disposal of well installation and decontamination fluids.
2. Arrange with the drilling contractor/client to provide a means of containment and disposal of fluids.

3.0 EQUIPMENT DECONTAMINATION

Prior to use and between each borehole location, drilling and sampling equipment must be decontaminated in accordance with Section 7.0 of the FSP.

4.0 LOCATION AND MARKING OF VAS/TEMPORARY MONITORING WELL SITES/FINAL VISUAL CHECK

The proposed investigative locations marked on the site plan are located and staked in the field. This should be completed several days prior to the drill rig arriving on site. Investigative locations are required for the completion of utility locates. Generally, VAS

or temporary monitoring well locations locations are strategically placed to assess site hydrogeologic conditions.

Once the final VAS or temporary monitoring well location has been selected and utility clearances are complete, one last visual check of the immediate area should be performed before drilling proceeds to confirm the locations of adjacent utilities (subsurface or overhead) and verify adequate clearance. If gravity sewers or conduits exist in the area, access manholes or chambers should be opened and the conduit/sewer alignments confirmed. Do not enter manholes unless confined space procedures are followed.

When possible, it is prudent to use a hand auger or post-hole digging equipment to a sufficient depth to confirm that there are no buried utilities or pipelines. This is particularly important in limited space sites where wells are being installed close to buried utilities. Alternatively, a Hydrovac truck can vacuum a large diameter hole to check for utilities, although soils collected this way may require containment on site. This procedure generally clears the area to the full diameter of the drilling equipment which will follow.

***Caution**: Do not assume that site plan details regarding pipe alignment/position are correct. Visually inspect pipe alignment when advancing boreholes near sewers. Be prepared to find additional piping if outdated plans are being used. If possible confirm pipe locations with on-site employees or a client representative.*

Investigative locations are selected primarily to provide a good geographical distribution across the site. Most often, the VAS or temporary monitoring well locations specified in the Work Plan are not pre-verified to confirm clearance from underground or overhead utilities, or to consider site-specific physical characteristics (e.g., traffic patterns, drainage patterns). Consequently, it is the Field Supervisor's responsibility to perform the following:

1. Select the exact location of each well consistent with the site and project requirements.
2. If a VAS or temporary monitoring well location must be relocated more than 20 feet (5.7 m) from the initially identified location, confirm the new location's suitability with the Project Coordinator.
3. Ensure all utilities have been cleared prior to initiating borehole advancement activities.

To the extent practical, wells should be located adjacent to permanent structures (e.g., fences, buildings) that offer some form of protection and a reference point for

future identification. Wells located in high traffic areas or road allowances or low-lying wet areas are undesirable, but may be unavoidable.

5.0 PROCEDURES FOR VERTICAL AQUIFER SAMPLING/TEMPORARY MONITORING WELL INSTALLATION AND SAMPLING BY GEOPROBE

The direct push procedure will use the Geoprobe, as follows:

1. The direct push drill rig will advance the borehole using methods consistent with ASTM Standard D6724-04 (Appendix J-H-4 of the FSP).
2. The direct push borehole will be advanced from ground surface to the top five feet of shallow groundwater. Soil cores will be collected using Geoprobe® MacroCore® sampling techniques or equivalent. Soil cores will be collected throughout the entire length of the borehole.
3. Representative samples will be logged immediately after opening the acetate liner. Field measurements of undifferentiated VOCs will be conducted by placing representative soil samples into a closed sample container and allowing them to equilibrate. The VOCs in the headspace will then be measured by placing the wand of the PID into the headspace. Field calibration, preventative maintenance, and SOPs for the PID are contained in Section 6.0 of the FSP.
4. The soil core will be logged by CRA personnel and soils will be classified using the USCS in accordance with ASTM Method D-2488-06 (Appendix J-H-2). Soil stratigraphy will be described on an Overburden Stratigraphy Log, an example of which is in Appendix J-G of the FSP.
5. Following the field screening and logging of the soil stratigraphy at each borehole, the Geoprobe will be offset approximately 1 foot from the borehole in order to collect a groundwater sample while preventing drawdown.
6. VAS will be conducted beginning no deeper than 5 feet below the water table unless otherwise specified in the Work Plan. Temporary monitoring well sampling will be conducted within the top 5 feet of the water table unless otherwise specified in the Work Plan.
7. A pre-cleaned Geoprobe® SP16 groundwater sampler will be assembled as per manufacturer's operational procedure. A description of the Geoprobe® SP16 is provided in Section 1.0 of this SOP.
8. New 1/4-inch diameter tubing will be installed and attached to a peristaltic or bladder pump. Groundwater will be purged from the Geoprobe® SP16 groundwater sampler using the pump. A minimum of three to five screen point well volumes will be purged at the same rate as the low flow sampling prior to commencing stabilization monitoring. Field measurements of pH, conductivity,

turbidity, and temperature will be collected at approximate 5-minute intervals. If it is apparent that stabilization will not be achieved quickly, stabilization parameter measurements may be made at a greater time interval. Stabilization monitoring will be performed using a flow-through-cell. All field measurements will be recorded in the field book.

The groundwater will be considered stable after a maximum of five well volumes are removed or when three successive readings for pH, specific conductance, turbidity, and temperature agree within the following limits:

- pH: ± 0.1 pH unit
- Specific conductance: ± 3% (temperature corrected)
- Temperature: ± 1.0 °C
- Turbidity: or < 5 NTU

9. Once field parameters have stabilized, groundwater samples will be collected directly from the discharge line in laboratory-supplied, analyte-specific sample containers and preserved according to laboratory requirements. Groundwater samples collected for VOC analysis will be collected from the tubing before it reaches the pump head, by crimping the tubing, detaching it from the pump, and pouring the water into the vial.
10. VAS and temporary monitoring well samples will be analyzed for parameters detailed in the Work Plan. The Geoprobe® SP16 groundwater sampler will be decontaminated between samples following the procedures in Section 7.0 of the FSP.
11. Upon reaching the total depth of the VAS or temporary monitoring well location, the downhole equipment will be removed from the borehole and the borehole will be backfilled with pure bentonite slurry grout.
12. All downhole equipment such as drill rods and sample tools will be decontaminated as discussed in Section 7.0 of the FSP.
13. Drill cuttings and decontamination water will be managed as discussed in Section 8.0 of the FSP.

6.0 SOIL SAMPLE COLLECTION FROM GEOPROBE DRILLING CORES

When borehole drilling, the core sample retrieved from the borehole is considered a discrete grab sample that has been taken from one sampling location, as long as both the stratigraphy of the entire sample and the level of contamination are consistent over the length of the core sample. If a single core sample contains soils from two different

stratigraphic units, the soils from each of these stratigraphic units are considered separate discrete grab samples.

If a single core sample contains soils from a single stratigraphic unit, but visual observation indicated that some of the soil was heavily impacted with contaminants, while the rest of the soil was only lightly impacted, then the soils representing each of the two levels of contamination are considered two separate discrete grab samples.

If required, representative soil samples will be collected from the drilling cores in accordance with the following procedures.

i. Once removed to the ground surface, open the discrete soil sampler by removing the cutting shoe, and extract the soil liner (with recovered soil) from the sampler body.

ii. Place the soil liner into a holder and cut lengthwise (using a liner knife) to expose the collected soil core.

iii. Perform PID screening for organic vapors and record readings.

iv. Measure length of sample and record as the recovered length.

v. Representative soil samples will be collected from Geoprobe drilling cores using a pre-cleaned stainless steel trowel or other appropriate tool (e.g., spoons or push tube.

vi. Use a new pair of disposable gloves for each sample.

vii. Prior to use, for each sample, decontaminate all sampling tools as specified in the Work Plan or as described in Section J.7 of the FSP.

viii. Use a pre-cleaned sampling tool to remove the sample from the layer of exposed soil. For clayey or cohesive soils,

   a. Discard upper and lower ends of sample core (3 inches) if near the area to be sampled.
   b. Use a pre-cleaned stainless steel knife.
   c. Cut the portion of the core to be sampled longitudinally.
   d. With a sample spoon, remove soil from the center portion of the core and place in a pre-cleaned stainless steel bowl.
   e. Remove large stones and natural vegetative debris.
   f. Homogenize the soil and place directly into sample jars.

   For sandy or non-cohesive soils, as sandy soils have less cohesion than clayey soils, it is not easy to cut the core longitudinally to remove the center of the sample. Therefore, with a stainless steel spoon, scrape away surface soils which have likely contacted the sampler and then sample the center portion of the soil core.

Note: Samples for VOC analysis must not be homogenized. Collect soil samples for VOC analysis in En Core™ Samplers (refer to Appendix J-F-24 of the FSP). Completely fill the container. No air space (headspace) should remain in the sample container.

ix. Place the collected soil directly into a clean, pre-labeled sample jar and seal with a Teflon-lined cap. If a sample is to be split for duplicate analyses, first homogenize the soil in a pre-cleaned stainless steel bowl (with the exception of samples for VOC analysis, which shall be placed directly in the sample jar and not homogenized in order to prevent volatilization of the VOCs).

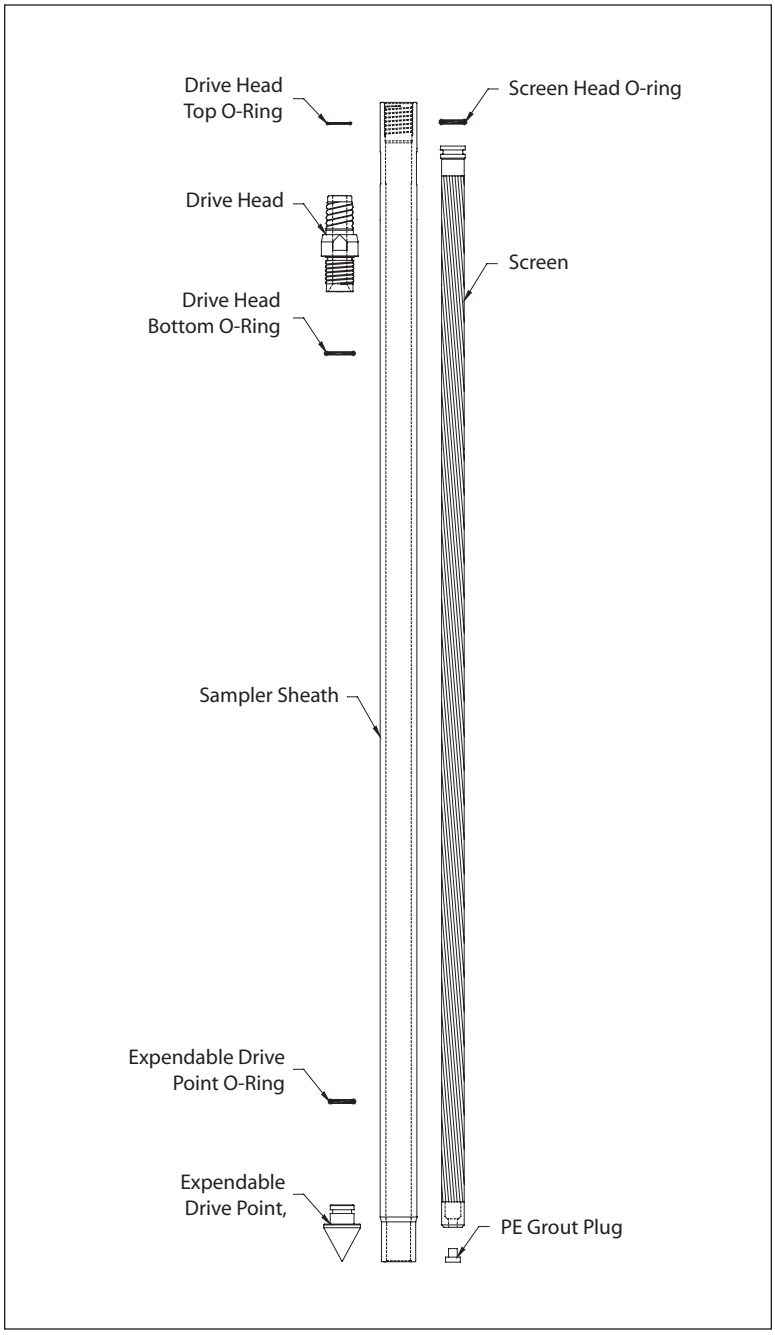
Note: Place all soil samples collected for chemical analysis immediately into a cooler with ice.

GEOPROBE® SCREEN POINT 16 GROUNDWATER SAMPLER

STANDARD OPERATING PROCEDURE

Technical Bulletin No. MK3142

PREPARED: November, 2006



GEOPROBE® SCREEN POINT 16 GROUNDWATER SAMPLER PARTS

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Screen Point 16 Groundwater Sampler is manufactured under U.S. Patent 5,612,498

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1.0 OBJECTIVE

The objective of this procedure is to drive a sealed stainless steel or PVC screen to depth, deploy the screen, obtain a representative water sample from the screen interval, and grout the probe hole during abandonment. The Screen Point 16 Groundwater Sampler enables the operator to conduct abandonment grouting that meets American Society for Testing and Materials (ASTM) Method D 5299 requirements for decommissioning wells and borings for environmental activities (ASTM 1993).

2.0 BACKGROUND

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and monitoring, soil conductivity and contaminant logging, grouting, and materials injection.

Screen Point 16 (SP16) Groundwater Sampler: A direct push device consisting of a PVC or stainless steel screen that is driven to depth within a sealed, steel sheath and then deployed for the collection of representative groundwater samples. The assembled SP16 Sampler is approximately 51.5 inches (1308 mm) long with an OD of 1.625 inches (41 mm). Upon deployment, up to 41 inches (1041 mm) of screen can be exposed to the formation. The Screen Point 16 Groundwater Sampler is designed for use with 1.5-inch probe rods and machines equipped with the more powerful GH60 Hydraulic Hammer. Operators with GH40 Series hammers may chose to use this sampler in soils where driving is difficult.

Rod Grip Pull System: An attachment mounted on the hydraulic hammer of a direct push machine which makes it possible to retract the tool string with extension rods or flexible tubing protruding from the top of the probe rods. The Rod Grip Pull System includes a pull block with rod grip jaws that are bolted directly to the machine. A removable handle assembly straddles the tool string while hooking onto the pull block to effectively grip the probe rods as the hammer is raised. A separate handle assembly is required for each probe rod diameter.

2.2 Discussion

In this procedure, the assembled Screen Point 16 Groundwater Sampler (Fig. 2.1A) is threaded onto the leading end of a Geoprobe® probe rod and advanced into the subsurface with a Geoprobe® direct push machine. Additional probe rods are added incrementally and advanced until the desired sampling interval is reached. While the sampler is advanced to depth, O-ring seals at each rod joint, the drive head, and the expendable drive point provide a watertight system. This system eliminates the threat of formation fluids entering the screen before deployment and assures sample integrity.

Once at the desired sampling interval, extension rods are sent downhole until the leading rod contacts the bottom of the sampler screen. The tool string is then retracted approximately 44 inches (1118 mm) while the screen is held in place with the extension rods (Fig. 2.1B). As the tool string is retracted, the expendable point is released from the sampler sheath. The tool string and sheath may be retracted the full length of the screen or as little as a few inches if a small sampling interval is desired.

There are three types of screens that can be used in the Screen Point 16 Groundwater Sampler. Two of the these, a stainless steel screen with a standard slot size of 0.004 inches (0.10 mm) and a PVC screen with a standard slot size of 0.010 inches (0.25 mm), are recovered with the tool string after sampling. The third screen is also manufactured from PVC with a standard slot size of 0.010 inches (0.25 mm), but is designed to be left downhole when sampling is complete. This disposable screen has an exposed screen length of approximately 43 inches (1092 mm). The two screens that are recovered with the sampler both have an exposed screen length of approximately 41 inches (1041 mm).

(continued on following page)

An O-ring on the head of the stainless steel screens maintains a seal at the top of the screen. As a result, any liquid entering the sampler during screen deployment must first pass through the screen. PVC screens do not require an O-ring because the tolerance between the screen head and sampler sheath is near that of the screen slot size.

The screens are constructed such that flexible tubing, a mini-bailer, or a small-diameter bladder pump can be inserted into the screen cavity. This makes direct sampling possible from anywhere within the saturated zone. A removable plug in the lower end of the screens allows the user to grout as the sampler is extracted for further use.

Groundwater samples can be obtained in a number of ways. A common method utilizes polyethylene (TB25L) or Teflon® (TB25T) tubing and a Check Valve Assembly (GW4210). The check valve (with check ball) is attached to one end of the tubing and inserted down the casing until it is immersed in groundwater. Water is pumped through the tubing and to the ground surface by oscillating the tubing up and down.

An alternative means of collecting groundwater samples is to attach a peristaltic or vacuum pump to the tubing. This method is limited in that water can be pumped to the surface from a maximum depth of approximately 26 feet (8 m). Another technique for groundwater sampling is to use a stainless steel Mini-Bailer Assembly (GW41). The mini-bailer is lowered down the inside of the casing below the water level where it fills with water and is then retrieved from the casing.

The latest option for collecting groundwater from the SP16 sampler is to utilize a Geoprobe® MB470 Series Mechanical Bladder Pump (MBP)*. The MBP may be used to meet requirements of the low-flow sampling protocol (Puls and Barcelona 1996, ASTM 2003). Through participation in a U.S. EPA Environmental Technology Verification study, it was confirmed that the MB470 can provide representative samples (EPA 2003).

**The Mechanical Bladder Pump is manufactured under U.S. Patent No. 6,877,965 issued April 12, 2005.*

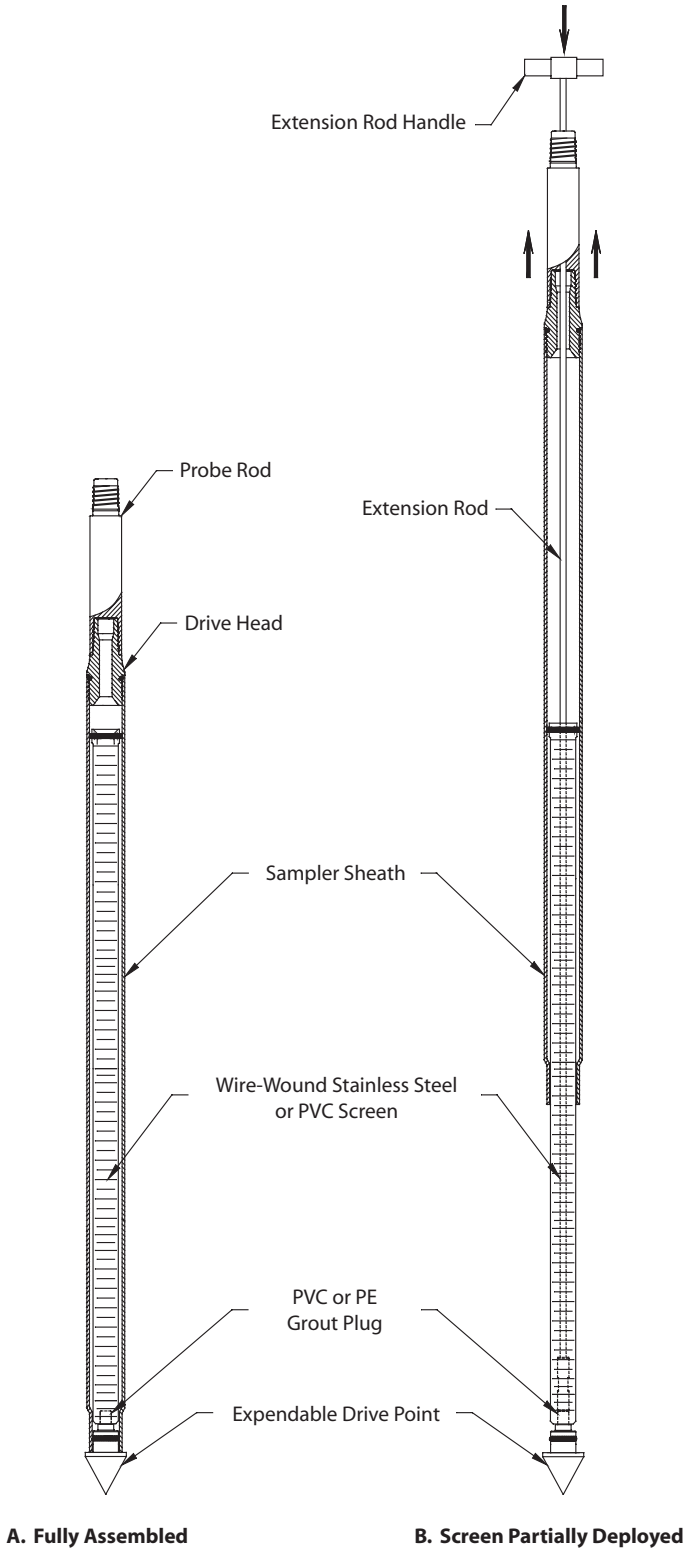


FIGURE 2.1
Screen Point 16 Groundwater Sampler

3.0 TOOLS AND EQUIPMENT

The following tools and equipment can be used to successfully recover representative groundwater samples with the Geoprobe® Screen Point 16 Groundwater Sampler. Refer to Figures 3.1 and 3.2 for identification of the specified parts. Tools are listed below for the most common SP16 / 1.5-inch probe rod configurations. Additional parts for optional rod sizes and accessories are listed in Appendix A.

SP16 Sampler Parts	**Part Number**
SP16 Sampler Sheath	15187
SP16 Drive Head, 0.5-inch bore, 1.5-inch rods*	18307
SP16 O-ring Service Kit, 1.5-inch rods *(includes 4 each of the O-ring packets below)*	15844
O-rings for Top of SP16 Drive Head, 1.5-inch rods only (Pkt. of 25)	*15389*
O-rings for Bottom of SP16 Drive Head (Pkt. of 25)	*13196*
O-rings for GW1520 Screen Head (Pkt. of 25)	*GW1520R*
O-rings for SP16 Expendable Drive Point (Pkt. of 25)	*GW1555R*
Screen, Wire-Wound Stainless Steel, 4-Slot*	GW1520
Grout Plugs, PE (Pkg. of 25)	GW1552K
Expendable Drive Points, steel, 1.625-inch OD (Pkg. of 25)*	GW1555K
Screen Point 16 Groundwater Sampler Kit, 1.5-inch Probe Rods *(includes 1 each of: 15187, 18307, 15844, GW1520, GW1535, GW1540, GW1555K, and GW1552K)*	15770

Probe Rods and Probe Rod Accessories	**Part Number**
Drive Cap, 1.5-inch probe rods, threadless, (for GH60 Hammer)	12787
Pull Cap, 1.5-inch probe rods	15090
Probe Rod, 1.5-inch x 60-inch*	11121

Extension Rods and Extension Rod Accessories	**Part Number**
Screen Push Adapter	GW1535
Grout Plug Push Adapter	GW1540
Extension Rod, 60-inch*	10073
Extension Rod Coupler	AT68
Extension Rod Handle	AT69
Extension Rod Jig	AT690
Extension Rod Quick Link Coupler, pin	AT695
Extension Rod Quick Link Coupler, box	AT696

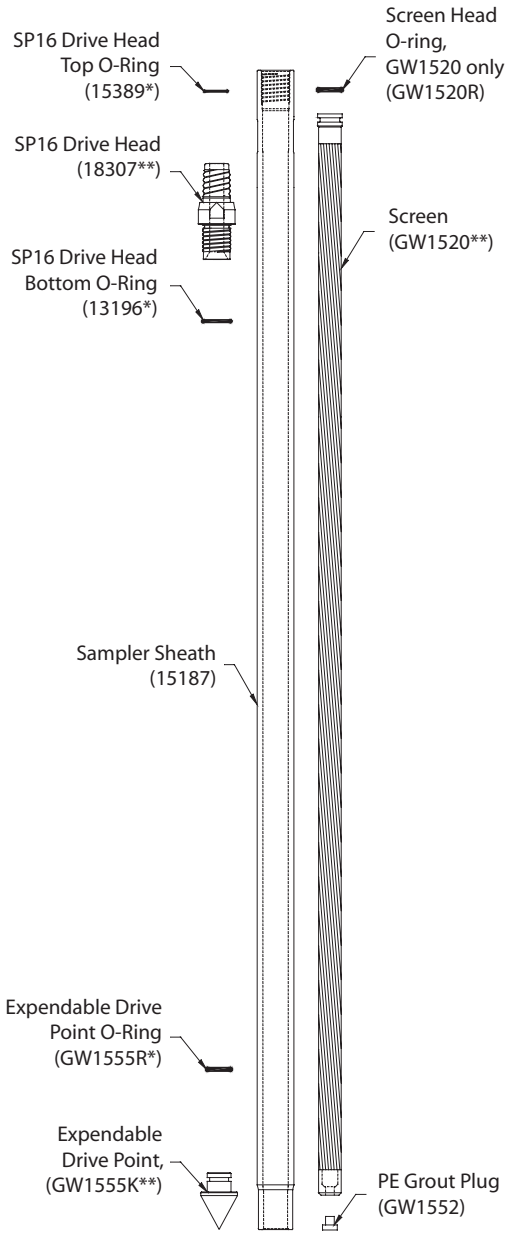
Grout Accessories	**Part Number**
Grout Nozzle, for 0.375-inch OD tubing	GW1545
High-Pressure Nylon Tubing, 0.375-inch OD / 0.25-inch ID, 100-ft. (30 m)	11633
Grout Machine, self-contained*	GS1000
Grout System Accossories Package, 1.5-inch rods	GS1015

Groundwater Purging and Sampling Accessories	**Part Number**
Polyethylene Tubing, 0.375-inch OD, 500 ft.*	TB25L
Check Valve Assembly, 0.375-inch OD Tubing*	GW4210
Water Level Meter, 0.438-inch OD Probe, 100 ft. cable*	GW2000
Mechanical Bladder Pump**	MB470
Mini Bailer Assembly, stainless steel	GW41

Additional Tools	**Part Number**
Adjustable Wrench, 6.0-inch	FA200
Adjustable Wrench, 10.0-inch	FA201
Pipe Wrenches	NA

* See Appendix A for additional tooling options.

** Refer to the Standard Operating Procedure (SOP) for the Mechanical Bladder Pump (Technical Bulletin No. MK3013) for additional tooling needs.



* Included in the SP16 O-ring Service Kit (15844)
** Part numbers show most common tool configuration. See Appendix A for alternative parts.

FIGURE 3.1
SP16 Sampler Parts

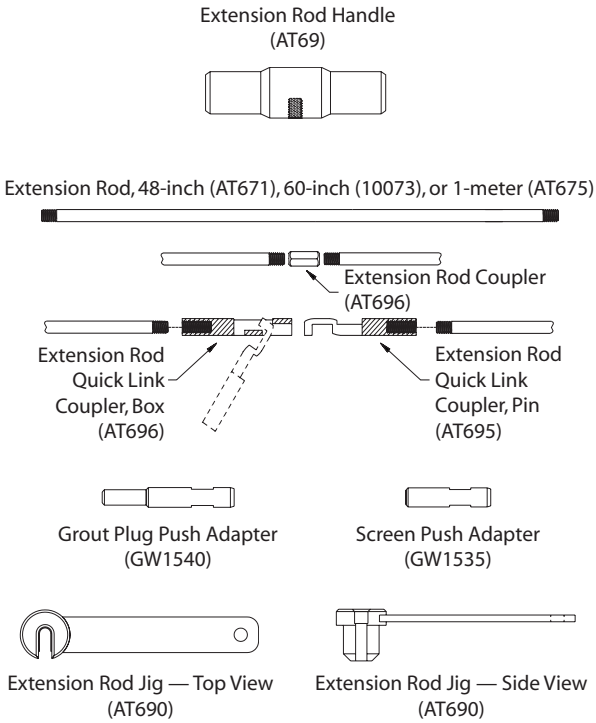


FIGURE 3.2
Geoprobe® Extension Rods and Accessories

4.0 OPERATION

4.1 Basic Operation

The SP16 sampler utilize a stainless steel or PVC screen which is encased in an alloy steel sampler sheath. An expendable drive point is placed in the lower end of the sheath while a drive head is attached to the top. O-rings on the drive head and expendable point provide a watertight sheath which keeps contaminants out of the system as the sampler is driven to depth.

Once the sampling interval is reached, extension rods equipped with a screen push adapter are inserted down the ID of the probe rods. The tool string is then retracted up to 44 inches (1118 mm) while the screen is held in place with the extension rods. The system is now ready for groundwater sampling. When sampling is complete, a removable plug in the bottom of the screen allows for grouting below the sampler as the tool string is retrieved.

4.2 Sampler Options

The Screen Point 15 and Screen Point 16 Groundwater Samplers are nearly identical. Subtle differences in the design of the SP16 sampler make it more durable than the earlier SP15 system. Operators of GH60-equipped machines should always utilize SP16 tooling. Operators of machines equipped with GH40 Series hammers may also choose SP16 tooling when sampling in difficult probing conditions.

A 1.75-inch OD Expendable Drive Point (17066K) and Disposable PVC Screen (16089) provide two useful options for the SP16 sampler. The 1.75-inch drive point may be used when soil conditions make it difficult to remove the sampler after driving to depth. The disposable PVC screen may be left downhole after sampling (when regulations permit) to eliminate the time required for screen decontamination.

4.3 Decontamination

In order to collect representative groundwater samples, all sampler parts must be thoroughly cleaned before and after each use. Scrub all metal parts using a stiff brush and a nonphosphate soap solution. Steam cleaning may be substituted for hand-washing if available. Rinse with distilled water and allow to air-dry before assembly.

4.4 SP16 Sampler Assembly (Figure 4.1)

Part numbers are listed for a standard SP16 sampler using 1.5-inch probe rods. Refer to Page 6 for screen and drive head alternatives.

1. Place an O-ring on a steel expendable drive point (GW1555K). Firmly seat the expendable point in the necked end of a sampler sheath (15187).

2. Install a PE Grout Plug (GW1552) in the bottom end of a Wire-wound Stainless Steel Screen (GW1520). Place a GW1520R O-ring in the groove on the top end of the screen.

3. Slide the screen inside of the sampler sheath with the grout plug toward the bottom of the sampler. Ensure that the expendable point was not displaced by the screen.

4. Install a bottom O-ring (13196) on a Drive Head (18307 or 15188). Thread the drive head into the sampler sheath using an adjustable wrench if necessary to ensure complete engagement of the threads. Attach a Drive Cap (12787 or 15590) to the top of the drive head.

   NOTE: The 18307 drive head should be used whenever possible as the smaller 0.5-inch ID provides a greater material cross-section for increased durability.

Sampler assembly is complete.

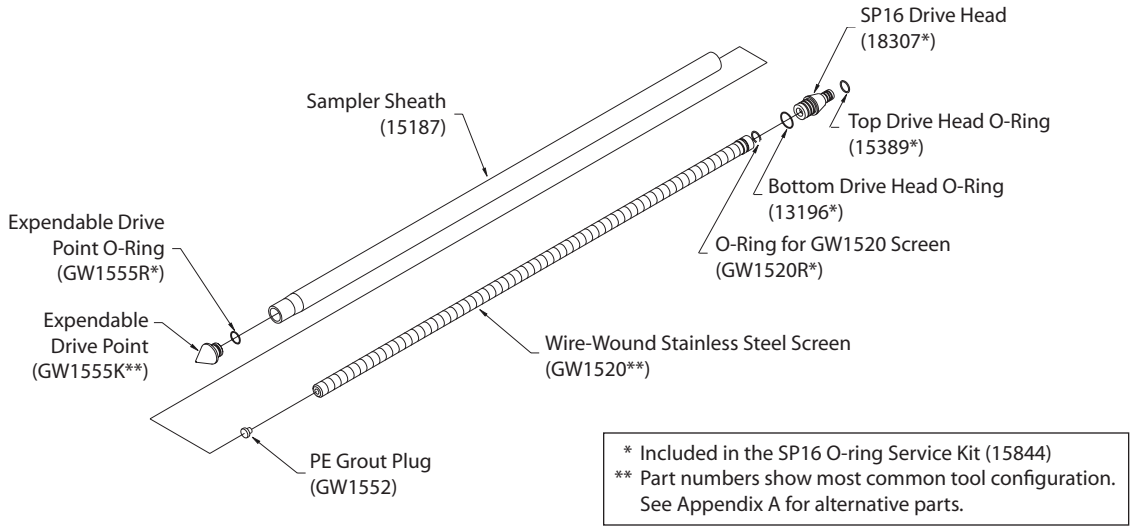


FIGURE 4.1
Screen Point 16 Groundwater Sampler Assembly

4.5 Advancing the SP16 Sampler

To provide adequate room for screen deployment with the Rod Grip Pull System, the probe derrick should be extended a little over halfway out of the carrier vehicle when positioning for operation.

1. Begin by placing the assembled sampler (Fig. 2.1.A) in the driving position beneath the hydraulic hammer of the direct push machine as shown in Figure 4.2.

2. Advance the sampler with the throttle control at slow speed for the first few feet to ensure that the sampler is aligned properly. Switch to fast speed for the remainder of the probe stroke.

3. Completely raise the hammer assembly. Remove the drive cap and place an O-ring in the top groove of the drive head. Distilled water may be used to lubricate the O-ring if needed.

   Add a probe rod (length to be determined by operator) and reattach the drive cap to the rod string. Drive the sampler the entire length of the new rod with the throttle control at fast speed.

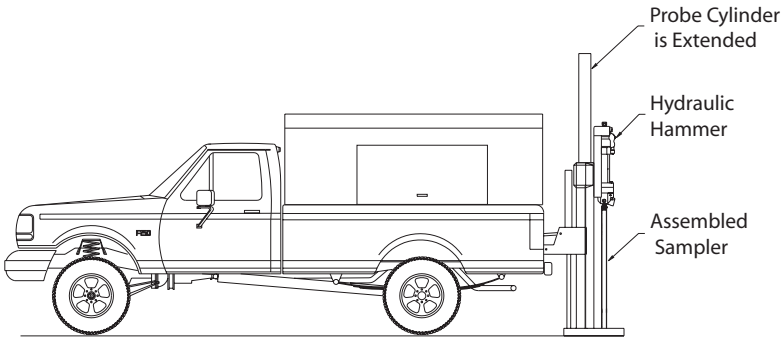


FIGURE 4.2
Screen Point 16 Groundwater Sampler in Driving Position

4. Repeat Step 3 until the desired sampling interval is reached. Approximately 12 inches (305 mm) of the last probe rod must extend above the ground surface to allow attachment of the puller assembly. A 12-inch (305 mm) rod may be added if the tool string is over-driven.

5. Remove the drive cap and retract the probe derrick away from the tool string.

4.6 Screen Deployment

1. Thread a screen push adapter (GW1535) on an extension rod of suitable length (AT671, 10073, or AT675). Attach a threaded coupler (AT68) to the other end of the extension rod. Lower the extension rod inside of the probe rod taking care not to drop it down the tool string. An extension rod jig (AT690) may be used to hold the rods.

2. Add extension rods until the adapter contacts the bottom of the screen. To speed up this step, it is recommended that Extension Rod Quick Links (AT695 and AT696) are used at every other rod joint.

3. Ensure that at least 48 inches (1219 mm) of extension rod protrudes from the probe rod. Thread an extension rod handle (AT69) on the top extension rod.

4. Maneuver the probe assembly into position for pulling.

5. Raise (pull) the tool string while physically holding the screen in place with the extension rods (Fig. 4.3.B). A slight knock with the extension rod string will help to dislodge the expendable point and start the screen moving inside the sheath.

   Raise the hammer and tool string about 44 inches (1118 cm) if using a GW1520 or GW1530 screen. At this point the screen head will contact the necked portion of the sampler sheath (Fig. 4.3.C.) and the extension rods will rise with the probe rods. Use care when deploying a PVC screen so as not to break the screen when it contacts the bottom of the sampler sheath.

   The Disposable Screen (16089) will extend completely out of the sheath if the tool string is raised more than 45 inches (1143 mm). Measure and mark this distance on the top extension rod to avoid losing the screen during deployment.

6. Remove the rod grip handle, lower the hammer assembly, and retract the probe derrick. Remove the top extension rod (with handle) and top probe rod. Finally, extract all extension rods.

7. Groundwater samples can now be collected with a mini-bailer, peristaltic or vacuum pump, tubing bottom check valve assembly, bladder pump, or other acceptable small diameter sampling device.

   When inserting tubing or a bladder pump down the rod string, ensure that it enters the screen interval. The leading end of the tubing or bladder pump will sometimes catch at the screen head giving the illusion that the bottom of the screen has been reached. An up-and-down motion combined with rotation helps move the tubing or bladder pump past the lip and into the screen.

4.7 Abandonment Grouting for GW1520 and GW1530 Screens

The SP16 Sampler can meet ASTM D 5299 requirements for abandoning environmental wells or borings when grouting is conducted properly. A removable grout plug makes it possible to deploy tubing through the bottom of GW1520 and GW1530 screens. A GS500 or GS1000 Grout Machine is then used to pump grout into the open probe hole as the sampler is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations.

1. Maneuver the probe assembly into position for pulling. Attach the rod grip puller to the top probe rod. Raise the tool string approximately 4 to 6 inches (102 to 152 cm) to allow removal of the grout plug.

2. Thread the Grout Plug Push Adapter (GW1540) onto an extension rod. Insert the adapter and extension rod inside the probe rod string. Add extension rods until the adapter contacts the grout plug at the bottom of the screen. Attach the handle to the top extension rod. When the extension rods are slightly raised and lowered, a relatively soft rebound should be felt as the adapter contacts the grout plug. This is especially true when using a PVC screen.

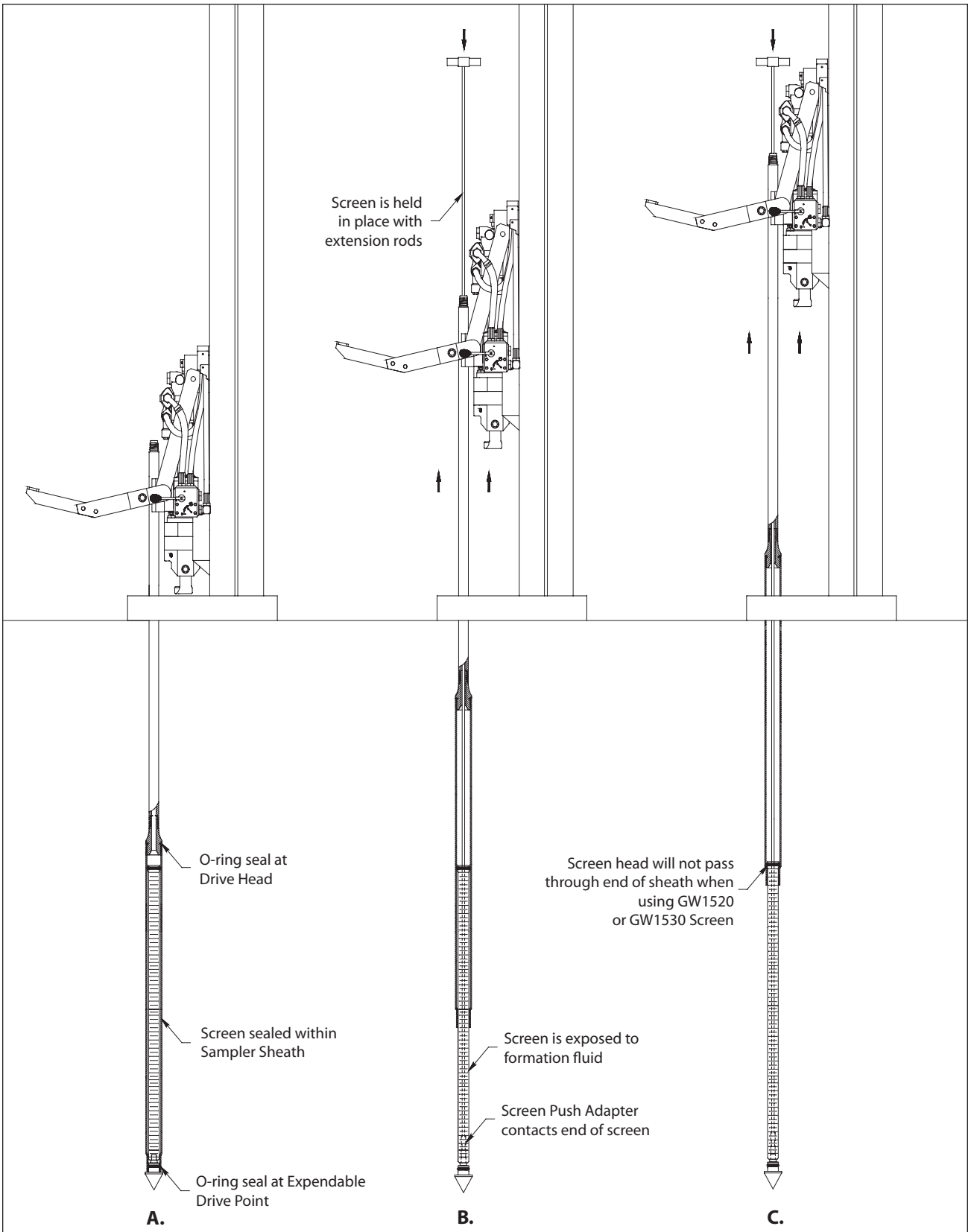


FIGURE 4.3
Screen Deployment for SP16 Sampler

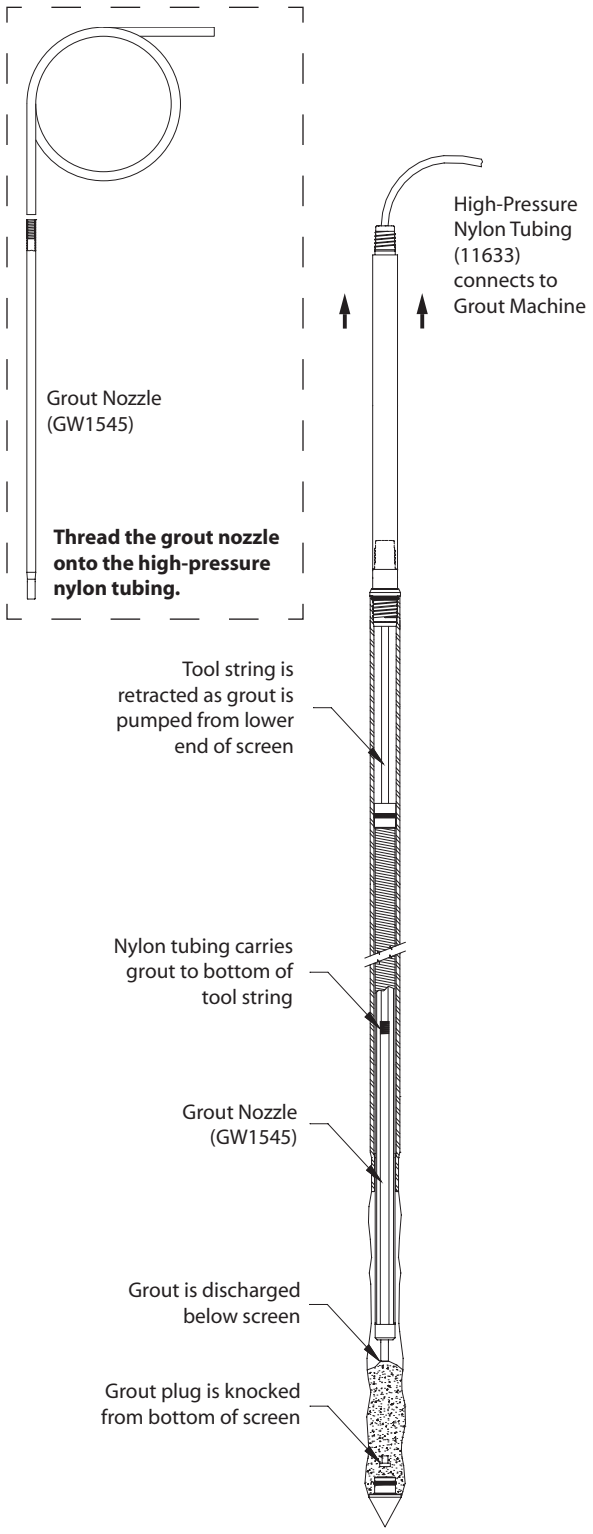


FIGURE 4.4
Grouting the SP16 Sampler With a GW1520 or GW1530 Screen

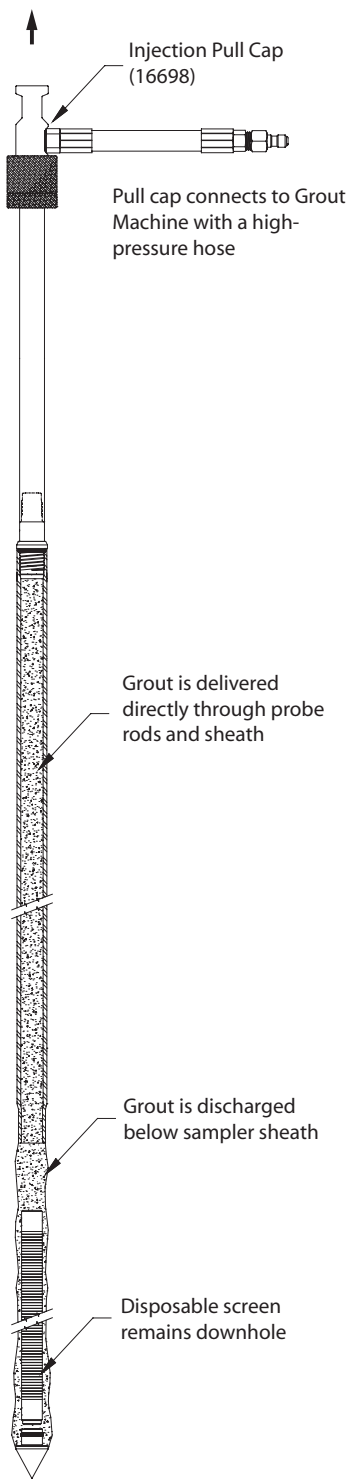


FIGURE 4.5
Grouting the SP16 Sampler With a 16089 Disposable Screen

3. Place a mark on the extension rod even with the top of the probe rod. Apply downward pressure on the extension rods and push the grout plug out of the screen. The mark placed on the extension rod should now be below the top of the probe rod. Remove all extension rods.

   Note: When working with a stainless steel screen, it may be necessary to raise and quickly lower the extension rods to jar the grout plug free. When the plug is successfully removed, a metal-on-metal sensation may be noted as the extension rods are gently "bounced" within the probe rods.

4. A Grout Nozzle (GW1545) is now connected to High-Pressure Nylon Tubing (11633) and inserted down through the probe rods to the bottom of the screen (Fig. 4.4). It may be necessary to pump a small amount of clean water through the tubing during deployment to jet out sediments that settled in the bottom of the screen. Resistance will sometimes be felt as the grout nozzle passes through the drive head. Rotate the tubing while moving it up-and-down to ensure that the nozzle has reached the bottom of the screen and is not hung up on the drive head.

   Note: *All probe rods remain strung on the tubing as the tool string is pulled. Provide extra tubing length to allow sufficient room to lay the rods on the ground as they are removed. An additional 20 feet is generally enough.*

5. Operate the grout pump while pulling the first rod with the rod grip pull system. Coordinate pumping and pulling rates so that grout fills the void left by the sampler. After pulling the first rod, release the rod grip handle, fully lower the hammer, and regrip the tool string. Unthread the top probe and slide it over the tubing placing it on the ground near the end of the tubing.

6. Repeat Step 5 until the sampler is retrieved. Do not bend or kink the tubing when pulling and laying out the probe rods. Sharp bends create weak spots in the tubing which may burst when pumping grout. Remember to operate the grout pump only when pulling the rod string. The probe hole is thus filled with grout from the bottom up as the rods are extracted.

7. Promptly clean all probe rods and sampler parts before the grout sets up and clogs the equipment.

4.8 Abandonment Grouting for the 16089 Disposable Screen

ASTM D 5299 requirements can also be met for the SP16 samplers when using the 16089 disposable screen. Because the screen remains downhole after sampling, the operator may choose either to deliver grout to the bottom of the tool string with nylon tubing or pump grout directly through the probe rods using an Injection Pull Cap (16698). A GS500 or GS1000 Grout Machine is needed to pump grout into the open probe hole as the sampler is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations.

1. Maneuver the probe assembly into position for pulling with the rod grip puller.

2. Thread the screen push adapter onto an extension rod. Insert the adapter and extension rod inside the probe rod string. Add extension rods until the adapter contacts the bottom of the screen. Attach the handle to the top extension rod.

3. The disposable screen must be extended at least 46 inches (1168 mm) to clear the bottom of the sampler sheath. Considering the length of screen deployed in Section 4.7, determine the remaining distance required to fully extend the screen from the sheath. Mark this distance on the top extension rod.

4. Pull the tool string up to the mark on the top extension rod while holding the disposable screen in place.

   The screen is now fully deployed and the sampler is ready for abandonment grouting. Apply grout to the bottom of the tool string during retrieval using either flexible tubing (as described in Section 4.7) or an injection pull cap (Fig. 4.5). This section continues with a description of grouting with a pull cap.

5. Remove the rod grip handle and maneuver the probe assembly directly over the tool string. Thread an Injection Pull Cap (16698) onto the top probe rod and close the hammer pull latch over the top of the pull cap.

6. Connect the pull cap to a Geoprobe® grout machine using a high-pressure grout hose.

7. Operate the pump to fill the entire tool string with grout. When a sufficient volume has been pumped to fill the tool string, begin pulling the rods and sampler while continuing to operate the grout pump. Considering the known pump volume and sampler cross-section, time tooling withdrawal to slightly "overpump" grout into the subsurface. This will ensure that all voids are filled during sampler retrieval.

   The grouting process can lubricate the probe hole sufficiently to cause the tool string to slide back downhole when disconnected from the pull cap. Prevent this by withdrawing the tool string with the rod grip puller while maintaining a connection to the grout machine with the pull cap.

4.9 Retrieving the Screen Point 16 Sampler

If grouting is not required, the Screen Point 16 Sampler can be retrieved by pulling the probe rods as with most other Geoprobe® applications. The Rod Grip Pull System should be used for this process as it allows the operator to remove rods without completely releasing the tool string. This avoids having the probe rods fall back downhole when released during the pulling procedure. A standard Pull Cap (15164) may still be used if preferred. Refer to the Owner's Manual for your Geoprobe® direct push machine for specific instructions on pulling the tool string.

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Appendix A
ALTERNATIVE PARTS

The following parts are available to meet unique soil conditions. See section 3.0 for a complete listing of the common tool configurations for the Geoprobe® Screen Point 16 Groundwater Sampler.

SP16 Sampler Parts and Accessories	**Part Number**
SP16 Drive Head, 0.625-inch bore, 1.5-inch rods	15188
Expendable Drive Points, aluminum, 1.625-inch OD (Pkg. of 25)	GW1555ALK
Expendable Drive Points, steel, 1.75-inch OD (Pkg. of 25)	17066K
Screen, PVC, 10-Slot	GW1530
Screen, Disposable, PVC, 10-Slot	16089

Groundwater Purging and Sampling Accessories	**Part Number**
Polyethylene Tubing, 0.25-inch OD, 500 ft.	TB17L
Polyethylene Tubing, 0.5-inch OD, 500 ft.	TB37L
Polyethylene Tubing, 0.625-inch OD, 50 ft.	TB50L
Check Valve Assembly, 0.25-inch OD Tubing	GW4240
Check Valve Assembly, 0.5-inch OD Tubing	GW4220
Check Valve Assembly, 0.625-inch OD Tubing	GW4230
Water Level Meter, 0.375-inch OD Probe, 100-ft. cable	GW2001
Water Level Meter, 0.438-inch OD Probe, 200-ft. cable	GW2002
Water Level Meter, 0.375-inch OD Probe, 200-ft. cable	GW2003
Water Level Meter, 0.438-inch OD Probe, 30-m cable	GW2005
Water Level Meter, 0.438-inch OD Probe, 60-m cable	GW2007
Water Level Meter, 0.375-inch OD Probe, 60-m cable	GE2008

Grouting Accessories	**Part Number**
Grout Machine, auxiliary-powered	GS500

Probe Rods, Extension Rods, and Accessories	**Part Number**
Probe Rod, 1.5-inch x 1-meter	17899
Probe Rod, 1.5-inch x 48-inch	13359
Drive Cap, 1.5-inch rods (for GH40 Series Hammer)	15590
Rod Grip Pull Handle, 1.5-inch Probe Rods (for GH40 Series Hammer)	GH1555
Extension Rod, 48-inch	AT671
Extension Rod, 1-meter	AT675

Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.

Geoprobe Systems®

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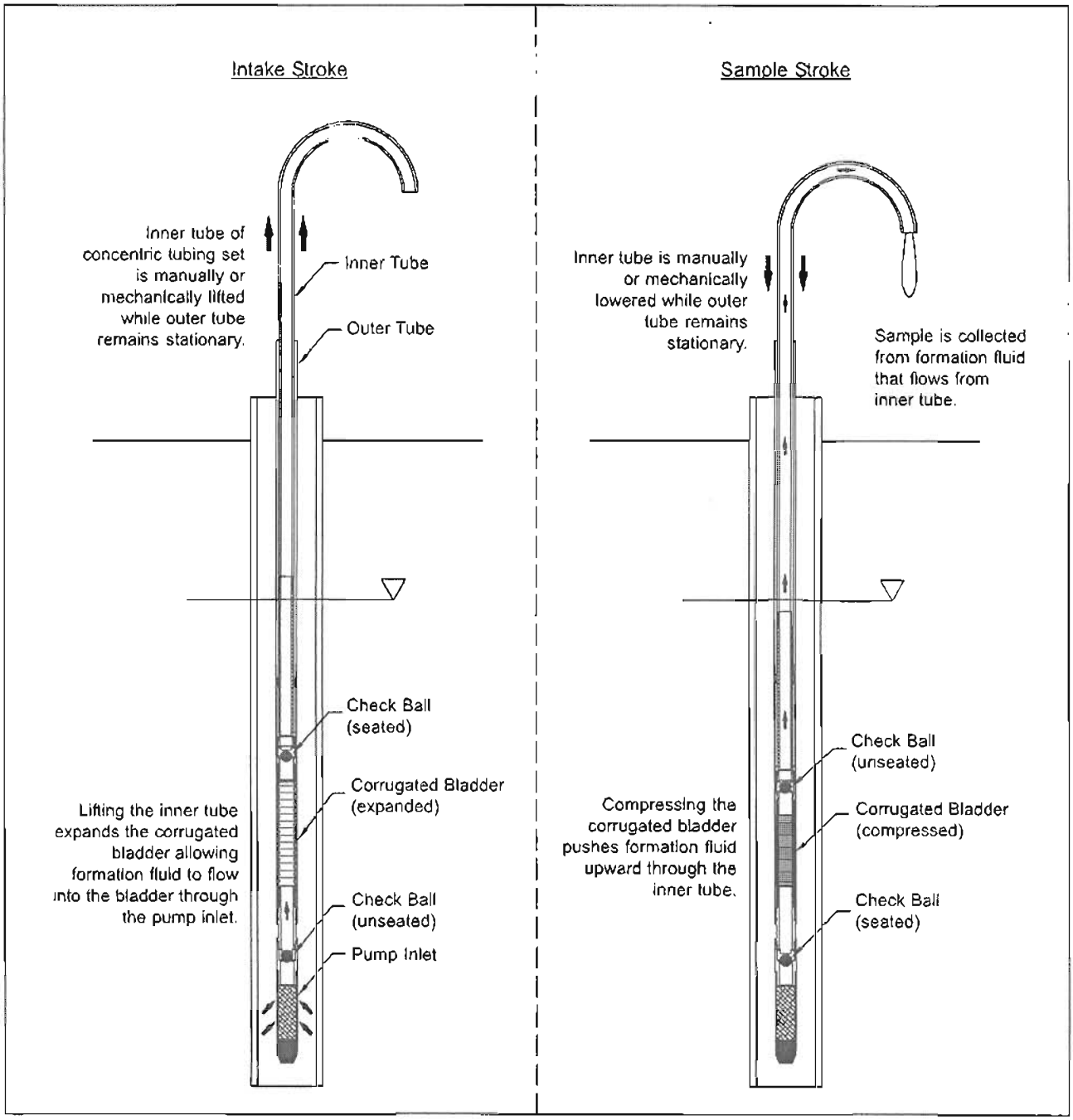
GEOPROBE® MODEL MB470 MECHANICAL BLADDER PUMP

STANDARD OPERATING PROCEDURE

Technical Bulletin No. MK3013

PREPARED: November, 2003
REVISED: July, 2006



INTAKE AND SAMPLE STROKES OF THE MB470 MECHANICAL BLADDER PUMP

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The Mechanical Bladder Pump is manufactured under
U.S. Patent No. 6,877,965 issued April 12, 2005.

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1.0 OBJECTIVE

The objective of this document is to provide guidance on how to collect a representative sample of the subsurface formation fluid utilizing the Geoprobe® Model MB470 Mechanical Bladder Pump.

2.0 BACKGROUND

2.1 Definitions

Geoprobe®*: A brand name of high quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and testing, soil conductivity and contaminant logging, grouting, and materials injection.
**Geoprobe® and Geoprobe Systems® are registered trademarks of Kejr, Inc., Salina, Kansas.*

MB470 Mechanical Bladder Pump (MBP):** A device for obtaining high-quality, low-turbidity samples from groundwater monitoring wells and direct push installed groundwater samplers as small as .5 inches (13 mm) inside diameter (ID). The MBP may be used to meet requirements of the low-flow sampling protocol (Puls and Barcelona 1996, ASTM 2003). Through participation in a U.S. EPA Environmental Technology Verification study, it was confirmed that the MB470 can provide representative samples (EPA 2003).
***The Mechanical Bladder Pump is manufactured under U.S. Patent No. 6,877,965 issued April 12, 2005.*

Within the MB470 pump body, a corrugated Teflon® fluorinated ethylene propylene (FEP) bladder is mechanically compressed and expanded to push groundwater to the surface through a concentric tubing set. Check valves above and below the bladder control flow direction. The outer tube of the concentric tubing set holds the pump body in place while the inner tube is used to actuate the bladder and transmit water to the surface. The pump body and internal components are made of stainless steel with an outside diameter (OD) of .47 inches (12 mm) and an overall length of 26.75 inches (679 mm) with an inlet screen assembly installed.

2.2 MBP System Components

The three basic components of the Model MB470 Mechanical Bladder Pump system are the pump, concentric tubing set, and actuator.

Pump

All pump components (Fig. 2.1) are made of stainless steel material with the exception of the three fluorosilicone O-rings and the Teflon® bladder.

Beginning at the downhole end of the pump, either a Bullet Nose Intake (P/N 20675) or Inlet Screen Assembly (P/N 20725) may be used as determined by project requirements. The screen assembly includes a 60 mesh wire screen with an actual screen length of 6 inches (152 mm). The bullet nose intake is open at the leading end and provides no filtering effect.

Above the intake/inlet, the pump body contains the corrugated bladder and check balls that physically move groundwater to the surface for purging and sampling. As the top of the bladder is extended, the expanding action of the bladder draws groundwater into the bladder through the intake/inlet. Compressing the bladder then pushes the groundwater up through the connected inner tube of the concentric tubing set. Check balls at the Upper and Lower Bladder Adapters (P/N 20679 and 20677) control groundwater flow through the bladder.

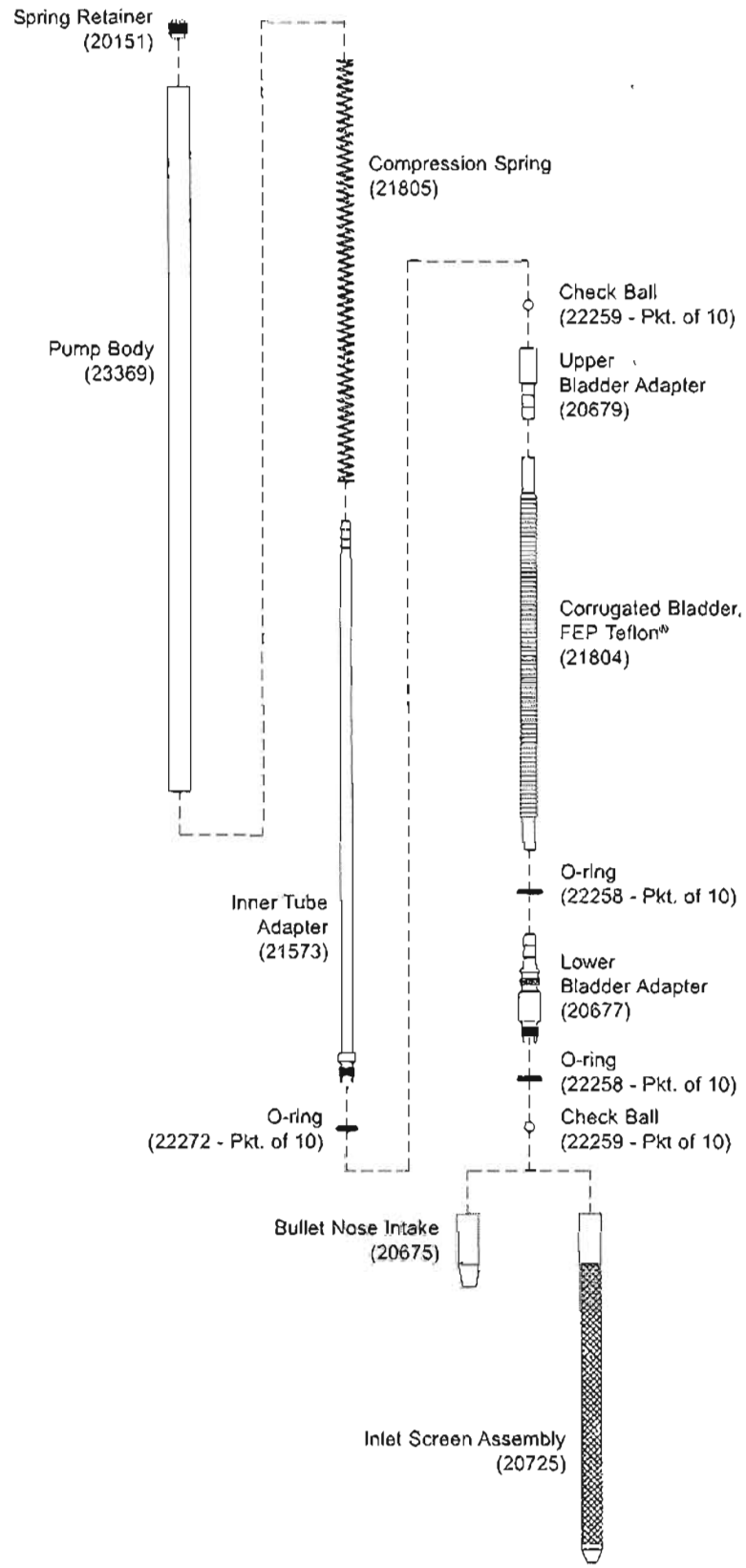

Figure 2.1: Components of MB470 Pump.

The lower end of the corrugated bladder is secured to the pump body by the Lower Bladder Adapter (P/N 20677). The top of the bladder is attached to the inner tube of the concentric tubing set by the Upper Bladder Adapter (P/N 20679) and Inner Tube Adapter (P/N 21573). During operation of the pump, the inner tube is raised and lowered to expand and contract the bladder to move formation fluid to ground surface.

Concentric Tubing Set

A concentric tubing set for the MB470 Mechanical Bladder Pump commonly consists of .19-inch (5 mm) ID / .25-inch (6 mm) OD Teflon® fluorinated ethylene propylene (FEP) tubing surrounded by .31-inch (8 mm) ID / .44-inch (11 mm) OD high-density polyethylene (HDPE) tubing. Where allowed by project requirements, other materials (e.g. low-density polyethylene (LDPE) tubing) may be utilized in place of the Teflon® inner tubing.

Available lengths for the concentric tubing set are 50 and 100 feet (15.2 and 30.5 m). Custom lengths may be assembled from 500-foot rolls of appropriate tubing sizes and materials, some of which are listed on Page 6.

Refer to the magnified view in Figure 2.2. The inner tube of the concentric tubing set is attached to the Inner Tube Adapter (P/N 21573) during assembly of the MB470 pump. The outer tube is then threaded inside the top end of the pump body. Once lowered down the sampler or monitoring well, the outer tube is held stationary either manually or by attachment to a mechanical actuator. The inner tube is raised and lowered by hand or through use of the mechanical actuator to expand and compress the pump bladder. Formation fluid is thus drawn into the pump bladder and then pushed to ground surface.

Actuator

Actuators provide the physical means of holding the outer tube of the concentric tubing set stationary while cycling the inner tube up-and-down. Actuator kits are available for manually or mechanically powering the MB470 pump.

For the manual actuator shown in Figure 2.2, the outer tube of the concentric tubing set is attached to the probe rods using two adapters. The inner tubing is raised and lowered by hand to obtain the groundwater sample. Refer to Section 4.4 for more actuator options.

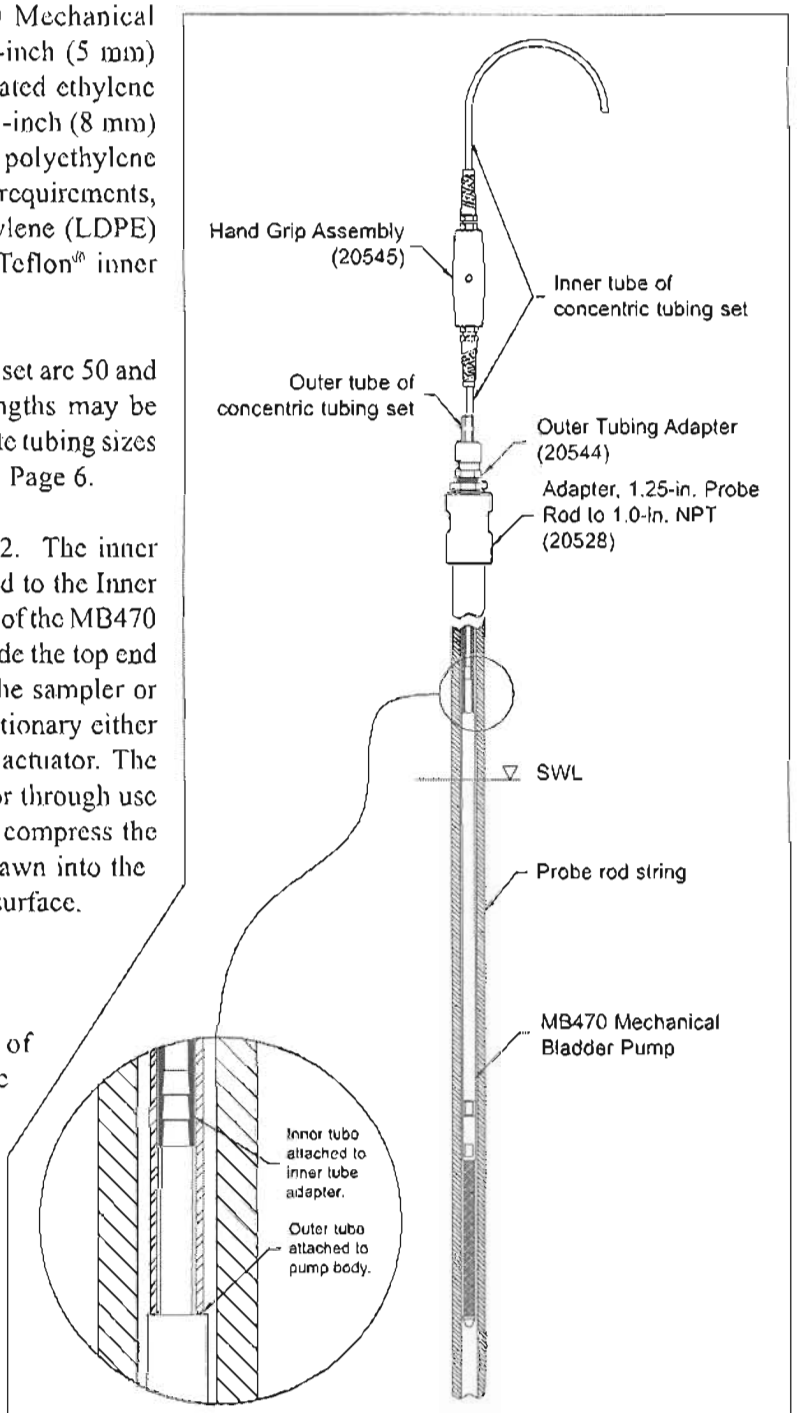

Figure 2.2: MB470 Mechanical Bladder Pump and manual actuator as used in a direct push installed groundwater sampler.

3.0 REQUIRED EQUIPMENT

The following equipment is required to collect representative groundwater samples using the Model MB470 Mechanical Bladder Pump. Refer to Figure 3.1 for identification of the specified parts.

Pump Components	Quantity	Part Number
Mechanical Bladder Pump	-1-	MB470
Service Parts Kit, for MB470 Pump	-1-	MB7500
Includes: O-ring Pick	-1-	AT102
Corrugated Bladder, Teflon* FEP	-3-	21804
Compression Spring, Stainless Steel (SS)	-1-	21805
O-rings for Lower Bladder Adapter (#5-585 Fluorosilicone), Pkg. of 10	-1-	22258
O-rings for Inner Tube Adapter (#010 Fluorosilicone), Pkg. of 10	-1-	22272
Check Balls (7/32-in. diameter), SS, Pkg. of 10	-1-	22259
MBP Assembly Tool	-1-	20456
MBP Cleaning Brush Kit	-1-	MB7300
MBP Assembly Tool	-1-	20456

Tubing Options	Quantity	Part Number
Concentric Tubing Set, HDPE (outer)/FEP (inner), .44-in. OD x 50-ft. length	Variable	MB5050
Concentric Tubing Set, HDPE/FEP, .44-in. OD - 100-ft. length	Variable	MB5100
Concentric Tubing Set, HDPE/LDPE, .44-in. OD - 50-ft. length	Variable	MB5051
Concentric Tubing Set, HDPE/LDPE, .44-in. OD - 100-ft. length	Variable	MB5101
Concentric Tubing Set, HDPE/PP, .44-in. OD - 50-ft. length	Variable	MB5052
Concentric Tubing Set, HDPE/PP, .44-in. OD - 100-ft. length	Variable	MB5102
LDPE Tubing, .19-in. ID x .25-in. OD - 100-ft. length	Variable	TB171L
LDPE Tubing, .19-in. ID x .25-in. OD - 500-ft. length	Variable	TB17L
Teflon* FEP Tubing, .19-in. ID x .25-in. OD - 50-ft. length	Variable	TB17T
Teflon* FEP Tubing, .19-in. ID x .25-in. OD - 100-ft. length	Variable	TB171T
Teflon* FEP Tubing, .19-in. ID x .25-in. OD - 500-ft. length	Variable	TB175T
PP Tubing, .17-in. ID x .25-in. OD - 50-ft. length	Variable	TB17P
PP Tubing, .17-in. ID x .25-in. OD - 100-ft. length	Variable	TB171P
HDPE Tubing, .31-in. ID x .44-in. OD - 50-ft. length	Variable	TB31H
HDPE Tubing, .31-in. ID x .44-in. OD - 100-ft. length	Variable	TB311H
HDPE Tubing, .31-in. ID x .44-in. OD - 500-ft. length	Variable	TB315H

Actuator Options	Quantity	Part Number
Manual Actuator Kit	-1-	MB7000
Includes: Hand Grip Assembly	-1-	20545
Outer Tubing Grip	-1-	22758
Outer Tubing Adapter	-1-	20544
Mechanical Actuator Assembly	-1-	MB6000
Electric Actuator Assembly, 12VDC	-1-	MB6120
Electric Actuator Kit, 12VDC	-1	MB6120K
Well Mount Kit (for use with MB6000)	-1-	MB7200

Adapters for Use with Actuators	Quantity	Part Number
MBP PVC Riser Adapter Kit	-1-	MB7100
Includes: PVC Extension, 1.0-in. NPT Pin x 1.0-in. NPT Pin - 12-in. Length	-1-	17560
PVC Coupling, 1.0-in. NPT Box x 1.0-in. NPT Box	-1-	21145
Adapter, 2.0-in. PVC to 1.0-in. NPT Pin	-1-	22759
O-rings for 2.0-in. PVC to 1.0-in. NPT Pin Adapter, pkg. of 25	-1-	22313
Adapter, 1.0-in. PVC to 1.0-in. NPT Pin	-1-	17558
O-rings for 1.0-in. PVC to 1.0-in. NPT Pin Adapter, pkg. of 25	-1-	13942
Adapter, 0.75-in. PVC to 17558 Adapter (0.75-in. PVC requires 2 adapters)	-1-	19424
O-rings for 0.75-in. PVC to 17558 Adapter, pkg. of 25	-1-	13196
Adapter, 0.5-in. PVC to 17558 Adapter (0.5-in. PVC requires 2 adapters)	-1-	17559
O-rings for 0.5-in. PVC to 17558 Adapter, pkg. of 25	-1-	GW1555R
Adapter, Geoprobe* 1.0-in. Probe Rod Pin to 1.0-in. NPT Pin	-1-	20527
Adapter, Geoprobe® 1.25-in. Probe Rod Pin to 1.0-in. NPT Pin	-1-	20528
Adapter, Geoprobe® 1.5-in. Probe Rod Pin to 1.0-in. NPT Pin	-1-	20529

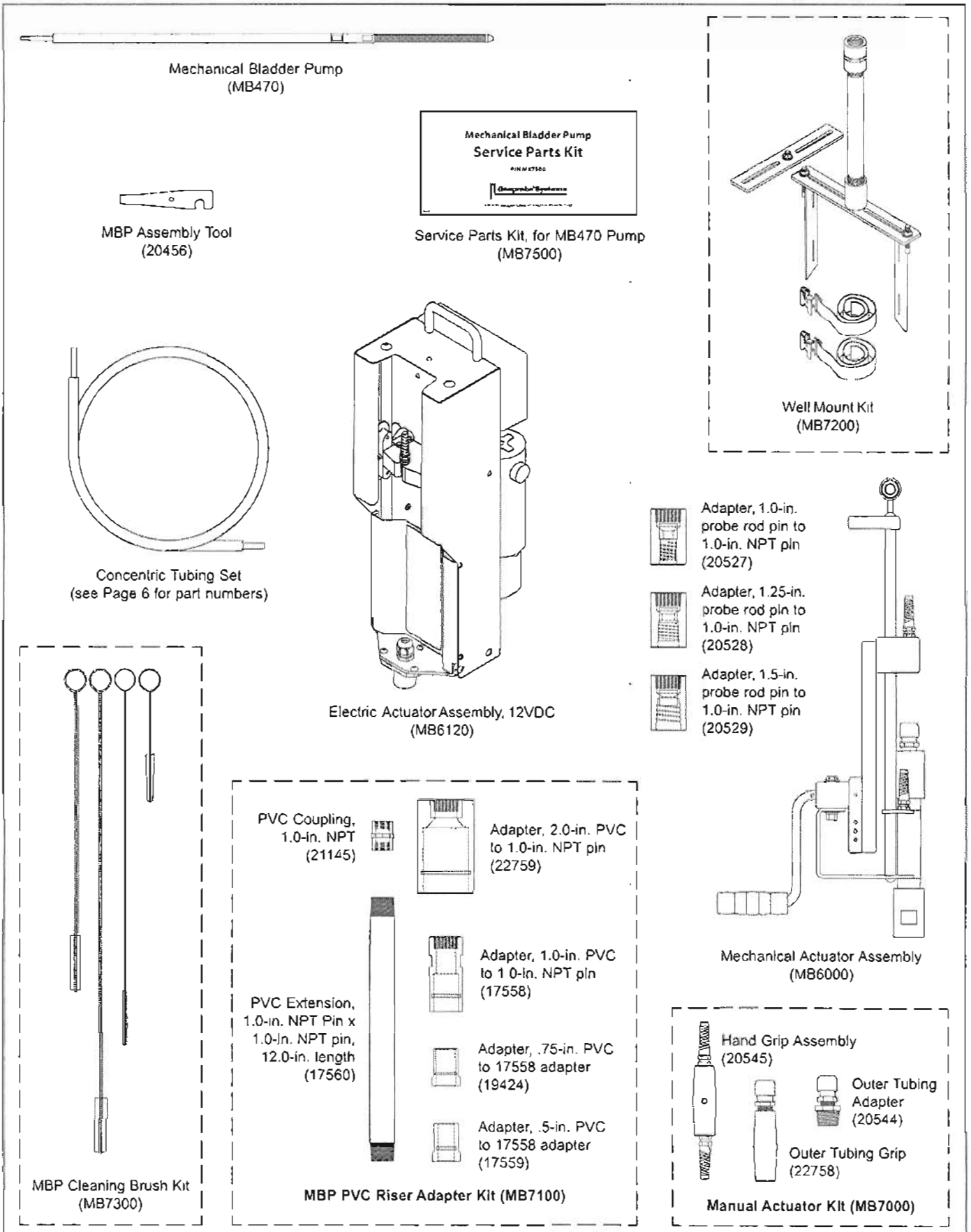

Figure 3.1: MB470 Mechanical Bladder Pump parts and accessories.

4.0 OPERATION

Use and operation of the MB470 Mechanical Bladder Pump may be divided into five main steps:

- *Assembling the Pump*
- *Selecting and installing the concentric tubing set*
- *Selecting and installing the actuator*
- *Purging and sampling*
- *Decontaminating the Pump*

4.1 Assembling the Pump

This section identifies the procedures for assembling the components of the MB470 Mechanical Bladder Pump and performing a leak check on the corrugated bladder. Refer to Figure 4.1 for parts identification.

1. Ensure that all metal parts are clean and free of burrs that may damage the pump threads or the corrugated bladder during assembly.

2. Install two fluorosilicone O-rings (22258) on the Lower Bladder Adapter (20677). Note that these are the larger of the two sizes of O-rings used with the MB470 pump.

3. Lubricate the O-ring of the lower bladder adapter and inside the Bullet Nose Intake (20675) with DI water. Place a Check Ball (22259) in the bullet nose intake and thread the intake onto the lower bladder adapter.

NOTE: The bullet nose intake is used here to make it easier to leak check the pump later in this procedure. After the leak check has been performed, the bullet nose intake may be replaced with a Screen Inlet Assembly (20725) if desired.

4. Install a fluorosilicone O-ring (22272) on the lower end of the Inner Tube Adapter (21573). Note that this is the smaller of the two sizes of O-rings used with the MB470 pump.

5. Lubricate the O-ring of the inner tube adapter and inside the Upper Bladder Adapter (20679) with DI water. Thread the upper bladder adapter onto the inner tube adapter.

NOTE: A check ball must be installed in the upper bladder adapter after performing the leak check in Step 7.

6. Install the Teflon® FEP Corrugated Bladder (21804):

   - The bladder should be installed with the corrugations pointing "up" (toward the upper bladder adapter/ inner tube adapter) as indicated in Figure 4.2.
   - Firmly push and rotate the lower cuff of the bladder over the barbed end of the lower bladder adapter.
   - Firmly push and rotate the upper cuff of the bladder over the barbed end of the upper bladder adapter.
   - Both ends of the bladder should be fully seated on the adapter barbs.

CAUTION: Although firmness is required during installation of the bladder, avoid crushing, kinking, or twisting the bladder corrugations to prevent damage.

7. Perform a leak check on the corrugated bladder before fully assembling the pump components to ensure that the bladder is free of defects. (Leak check procedure is given on opposite page.)

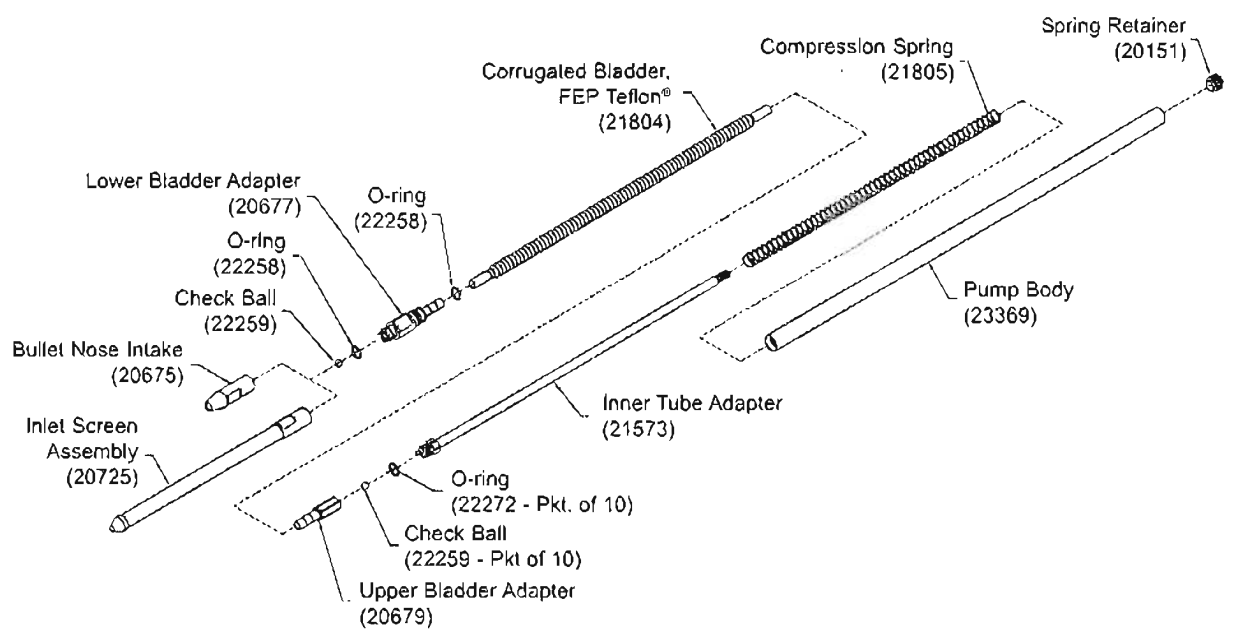

Figure 4.1: Isometric exploded view of the MB470 Mechanical Bladder Pump.

Leak check the corrugated bladder as follows:

- Completely submerge the bladder and lower end of the inner tube adapter in a clean beaker or small bucket of distilled or DI water.
- Firmly blow into the open end of the inner tube adapter. Leaks in the bladder or assembled parts will be indicated by bubbles.
- If leaks are found, replace the faulty O-ring(s) or bladder. Retest to ensure that all leakage has stopped.
- Once the pump has passed the leak test, unthread the upper bladder adapter from the inner tube adapter. Place a Check Ball (22259) in the upper bladder adapter and reinstall it in the inner tube adapter.
- Replace the bullet nose intake with an Inlet Screen Assembly (20725) if desired. Remember to include the check ball when installing the inlet screen.

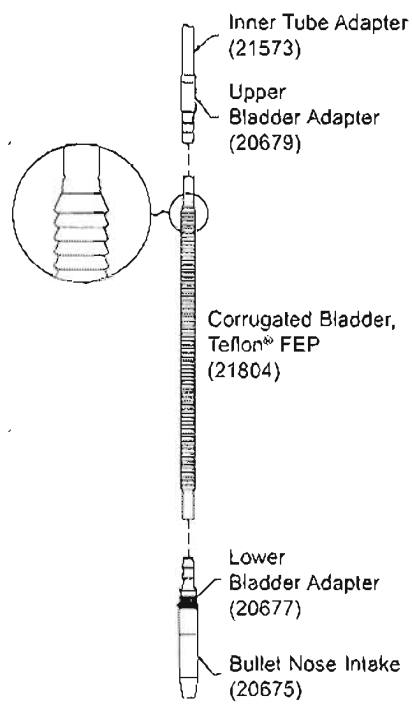

Figure 4.2: Bladder corrugations should point "up".

8. The Pump Body (23369) is internally threaded at each end. Threads run all the way to the end of the pump body at the upper end, but stop .25 inches (6 mm) from the end at the lower end of the pump body to permit an O-ring seal.

   Thread the Spring Retainer (20151) into the top of the pump body. Install the retainer with the slotted end out to allow use of a medium slotted screw driver or the MBP Assembly Tool (20456) to thread or unthread the retainer.

9. Place the Compression Spring (21805) over the top of the inner tube adapter. Slide the spring completely onto the adapter until it contacts the hex fitting.

10. Slide the lower end of the pump body over the top of the inner tube adapter and pump spring. The inner tube adapter will slip through the spring retainer and extend approximately 3 inches (75 mm) from the top of the pump body.

11. The lower bladder adapter is now threaded into the pump body to complete the assembly process.

- Lubricate the O-ring on the lower bladder adapter and inside the lower end of the pump body with DI water.
- Grasp the pump body with one hand and the lower bladder adapter with the other hand.
- Gently compress the spring and bladder into the pump body.
- Thread the lower bladder adapter into the pump body. Use care to avoid cutting or pinching the O-ring while threading the parts together. The O-ring will no longer be visible when the adapter is fully seated.

Assembly of the MB470 Mechanical Bladder Pump is now complete.

4.2 Selecting and Installing the Concentric Tubing Set

Selecting the Concentric Tubing Material and Length

The outer tube of the concentric tubing set commonly consists of .44-inch OD x .31-inch ID (11.2 mm x 7.9 mm) HDPE material. Inner tube material options are Teflon® FEP, LDPE, or PP. Teflon® FEP and LDPE tubing have dimensions of .25-inch OD x .19-inch ID (6.4 mm x 4.8 mm) while the PP tubing measures .25-inch OD x .17-inch ID (6.4 mm x 4.3 mm).

LDPE inner tubes are the least expensive option. The elasticity of this material may be excessive for deeper wells and in warm ambient conditions (summertime). Teflon® FEP inner tubes are less elastic and provide higher sample quality compared to LDPE due to the chemical properties of the two materials. Teflon® FEP also has a lower coefficient of friction for smoother actuation of the bladder and less resistance to operation, especially at greater depths. The main drawback of Teflon® FEP is its higher cost. PP inner tubes provide a compromise between LDPE and Teflon® FEP in that they are less elastic and provide higher sample quality than LDPE at a lower cost than Teflon® FEP.

While Teflon® FEP exhibits relatively good chemical inertness, it will absorb and desorb some volatile organic contaminants (Parker & Ranney 1998). Because of this, ambient groundwater should be purged through the pump and tubing system for a period of time to achieve equilibrium between the bladder and tubing and sample fluid. The period of time may vary for different volatile organic compounds (VOCs), but if low flow sampling (Puls and Barcelona 1996, ASTM 2003) is conducted, chemical equilibrium may be achieved by the time the monitored water quality parameters (DO, ORP, turbidity, etc.) have stabilized.

Preassembled concentric tubing sets are available from Geoprobe Systems® in lengths of 50 and 100 feet (15.2 and 30.5 m). The user may choose to assemble sets of custom lengths from separate rolls of inner and outer tubing in preparation for the sampling event or while on-site. Be careful to keep the tubing clean while inserting the inner tube into the outer tube.

When long tubing sets are required, it may be wise to use clean PVC riser pipe to protect the tubing during assembly. Simply thread PVC riser sections together, placing them on the shop floor or along the ground surface. Cap one end of the casing to keep dirt and debris out during assembly. Determine the length of the outer tube required and make the PVC casing about the same length. Slide the outer tube into the PVC casing and cut to the desired length. Slide the inner tube into the outer tube. Cut the inner tube three or more feet longer than the outer tube to complete the concentric tubing set.

Keep all tubing stored in clean airtight bags or containers so that dirt, dust, and cross contamination are not a concern or problem. No matter how clean the pump is, sample quality will suffer if the tubing is dirty. Be sure the tubing is of clean, quality material and is not marked with inks that may contribute to cross contamination.

Installing the Concentric Tubing Set on the MB470 Pump

The concentric tubing set is attached to the mechanical bladder pump by pushing the inner tube onto the hose barb on the end of the inner tube adapter and then threading the outer tube into the pump body.

1. Push the inner tube of the concentric tubing set onto the hose barb on the end of the inner tube adapter (Fig. 4.3). Fully seat the tube on the adapter such that the tube engages all three barbs. Take care not to kink or otherwise damage the tubing.

2. Before installing the outer tube, unthread the lower bladder adapter from the pump body and lay the partially disassembled pump on a clean, level surface. This step is recommended so that the bladder is not twisted or damaged as the outer tubing is installed.

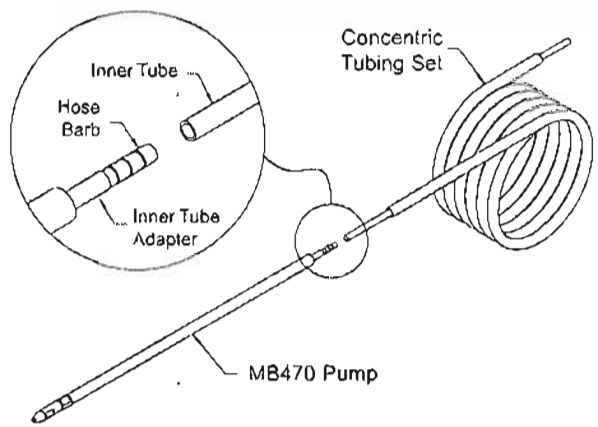

Figure 4.3: Fully seat the inner tube onto the hose barbs of the inner tube adapter.

3. Push and thread the outer tube into the top end of the pump body (Fig. 4.4). The outer tube should be threaded about 0.75 inches (19 mm) into the pump body until it butts against the spring retainer. Remember to take care not to kink or otherwise damage the tubing during installation.

4. Rotate the lower bladder adapter counterclockwise one or two revolutions to minimize torque on the bladder when threading the adapter into the pump body. Now reinstall the lower bladder adapter and inner tube adapter into the lower end of the pump body.

   The pump and tubing set are now assembled and ready for installation into the monitoring well or sampler.

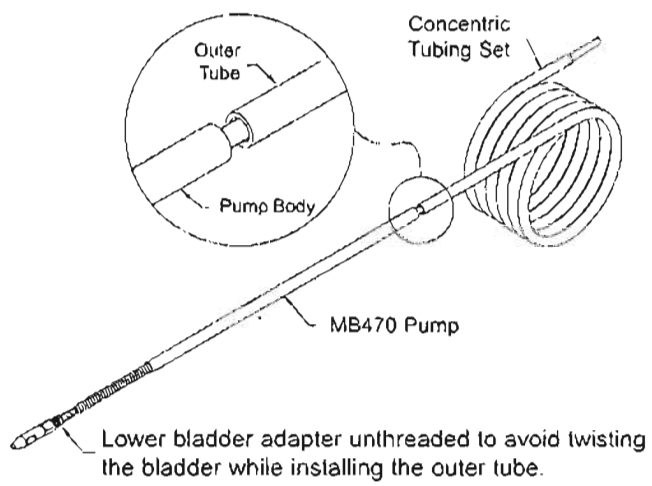

Figure 4.4: Thread the outer tube of the concentric tubing set into the pump body.

NOTE: Friction between the inner and outer tubes may make it difficult to attach the pump with the tubing set coiled. To overcome this problem, attach the pump while the concentric tubing is unrolled in the PVC riser sections as described at the bottom of Page 10.

The user may also choose to lower the concentric tubing set partway down the tool string or well, attach the pump to the exposed end of the tubing, retrieve the tubing set, and install the pump for purging or sampling. If this technique is used, **take great care to avoid dropping the tubing set down the well or tool string during attachment of the pump.**

4.3 Selecting and Installing the Actuator

Operating the mechanical bladder pump requires holding the outer tube of the concentric tubing set stationary while moving the inner tube up-and-down. Although this maneuver is possible by simply holding the outer tube in one hand and moving the inner tube with the other hand, an actuator makes operation of the pump significantly easier.

NOTE: The tubing set must be completely unrolled for the inner tube to slide freely within the outer tube.

This section identifies the available actuator options. Methods by which the actuators attach to the concentric tubing set and are installed on the monitoring well or tool string are also addressed.

Handheld Manual Actuator

The handheld actuator option is the first step above simply grasping the inner and outer tube by hand. With this option, a Hand Grip Assembly (20545) and Outer Tubing Grip (22758) are installed on the concentric tubing set (Fig. 4.5). Sampling or purging is accomplished by physically holding the outer tubing grip in one hand while raising and lowering the hand grip assembly with the other hand. A handheld actuator may be used to purge or collect samples through probe rods from a groundwater sampler as well as from a permanent monitoring well.

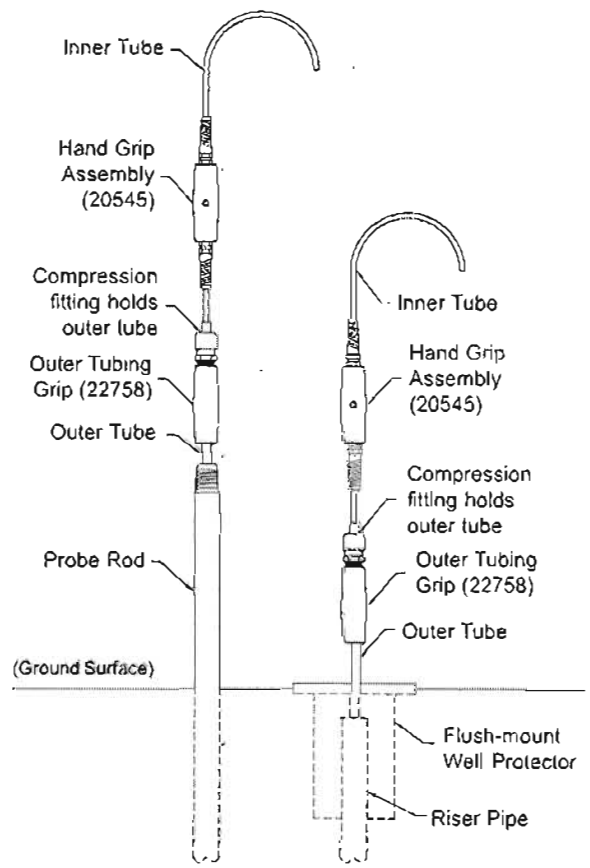

Figure 4.5: Handheld actuator parts for use with groundwater samplers and monitoring wells.

Installation of the handheld actuator is described below.

1. Determine the depth to which the pump inlet will be installed as measured from the top probe rod or riser pipe with a weighted tape or water lever meter.

2. The distance from the pump inlet to the top of the tool string or riser pipe (from Step 1) may now be marked on the outer tube. Obtain an assembled MB470 Mechanical Bladder Pump (Section 4.1) with a concentric tubing set installed as instructed in Section 4.2. Beginning from the pump inlet, measure the appropriate distance along the outer tube and mark it with electrical tape or a suitable marker. The tubing set will be installed such that this mark is aligned with the top of the probe rods or riser.

3. Leading with the end opposite the compression fitting, slide the outer tubing grip over the top end of the tubing set. It may be necessary to loosen the fitting slightly (Fig. 4.6) to allow installation.

4. Position the grip with the lower end even with, or slightly above the line marked on the outer tube in Step 2. The specific location of the grip should be determined by operator preference. The important thing is that the pump inlet is maintained at the appropriate level during sampling as indicated by the mark on the outer tube.

5. Secure the grip to the outer tube by tightening the large nut of the compression fitting (Fig. 4.6) until it is "hand tight". Do not overtighten as this may damage the plastic fitting.

6. Carefully cut off the excess outer tube leaving approximately .25 inches (6 mm) above the compression fitting. (Note that the inner tube is not cut at this location). Now measure and cut the inner tube leaving it approximately 3 feet (1 m) longer than the outer tube.

7. Slide the hand grip assembly over the inner tube and position it 1-2 inches (25-51 mm) above the outer tubing grip as shown in Figure 4.6. It may be necessary to first loosen the two compression fittings to allow installation over the inner tube.

8. Secure the hand grip by tightening the two compression fittings. Take care not to overtighten and damage the fittings. Also avoid kinking the inner tube while completing this step

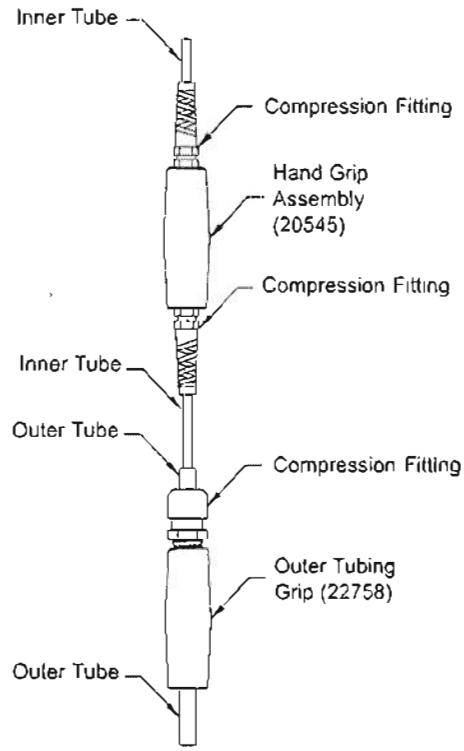

Figure 4.6: Close-up view of hand grip and outer tubing grip.

To operate the mechanical bladder pump with the handheld actuator, simply insert the pump into the probe rod string or monitoring well. Lower the pump and concentric tubing set until the mark on the outer tube (measured and marked previously in Step 2, Page 12) is aligned with the top of the probe rod string or well riser. Initiate pump flow by holding the outer tubing grip stationary with one hand while cycling the hand grip assembly up-and-down with the other hand. A pump stroke of up to approximately 6 inches (150 mm) is recommended.

Anchored Manual Actuator

The anchored actuator option is similar to the handheld actuator in that the mechanical bladder pump is cycled by physically raising and lowering the inner tube using the Hand Grip Assembly (20545). But while the handheld actuator requires a second hand to hold the outer tube, the anchored actuator option utilizes adapters to mechanically secure the outer tubing to the top probe rod or riser pipe as shown in Figure 4.7.

Installation of the mechanical bladder pump with the anchored actuator option is reviewed in this section for both probe rod and well riser applications.

1. The outer tube of the concentric tubing set is connected to the top probe rod or well riser using an Outer Tubing Adapter (20544) plus additional adapters as determined by the size of rod or riser onto which the actuator is to be installed.

   Referring to Table 4.1, select the appropriate adapter(s) for your size of probe rod or well riser. Illustrations and complete descriptions of the various adapters are presented in Table 4.2 and Figures 4.8 - 4.10. Note that .5-inch and 0.75-inch riser pipe each require two PVC adapters in addition to the outer tubing adapter.

2. Assemble the adapters by threading the outer tubing adapter into the probe rod or well riser adapter.

   As illustrated in Figure 4.8, two adapters are required to attach the outer tubing adapter to .5-inch and .75-inch riser pipe. After threading the outer tubing adapter into the 1.0-inch PVC to 1.0-inch NPT Adapter (17558), either a .5-inch PVC adapter (19424) or .75-inch PVC adapter (17559) is then installed in the remaining end of the 1.0-inch PVC adapter.

3. Determine the depth to which the pump inlet will be installed as measured from the top probe rod or riser pipe with a weighted tape or water lever meter.

4. The distance from the pump inlet to the top of the tool string or riser pipe (from Step 3) is now marked on the outer tube:

   Obtain an assembled MB470 Mechanical Bladder Pump (Section 4.1) with a concentric tubing set installed as instructed in Section 4.2. Beginning from the pump inlet, measure the appropriate distance along the outer tube and mark it with electrical tape or a suitable marker. The tubing set will be installed such that this mark is aligned with the top of the probe rods or riser.

5. Slide the assembled adapters (from Step 2) over the top end of the tubing set leading with the end opposite the compression fitting. See Figure 4.7 for adapter orientation. It may be necessary to loosen the compression fitting slightly to allow installation.

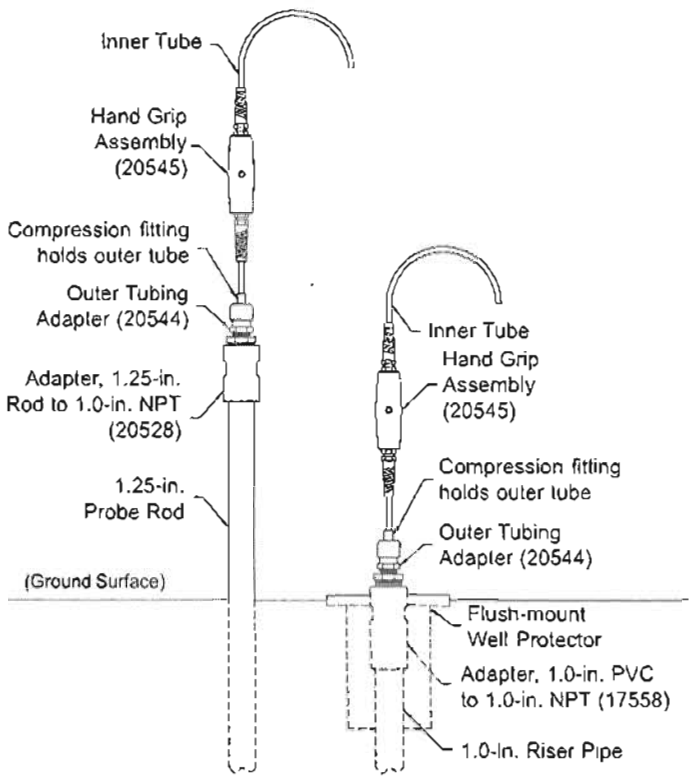

Figure 4.7: Manual actuator parts for use with groundwater samplers and monitoring wells. (Part numbers specified for 1.25-inch probe rods and 1.0-inch riser pipe.)

6. Position the adapters such that the line marked on the outer tube in Step 4 will be even with the top of the probe rod or well riser when the pump is installed on the tool string or riser.

7. Secure the adapters to the outer tube by tightening the large nut of the compression fitting (Fig. 4.7) until it is "hand tight". Do not overtighten as this may damage the plastic fitting.

Size	Probe Rod Adapters	Monitoring Well Riser Adapters
.5-inch	na	17559, 17558, and 20544
.75-inch	na	19424, 17558, and 20544
1.0-inch	20527 and 20544	17558 and 20544
1.25-inch	20528 and 20544	na
1.5-inch	20529 and 20544	na
2.0-inch	na	22759 and 20544

Table 4.1: Part numbers for the adapters required to attach the outer tube to various probe rods and PVC riser pipe.

8. Carefully cut off the excess outer tube leaving approximately .25 inches (6 mm) above the compression fitting. (Note that the inner tube is not cut at this location). Now measure and cut the inner tube leaving it approximately 3 feet (1 m) longer than the outer tube.

9. Slide the hand grip assembly over the inner tube and position it 1-2 inches (25-51 mm) above the outer tubing grip as shown previously in Figure 4.6. It may be necessary to first loosen the two compression fittings to allow installation over the inner tube.

10. Secure the hand grip by tightening the two compression fittings until they are hand tight. Do not overtighten the plastic fittings as damage may result.

Illustration	Part Number	Description
	20544	Outer Tubing Adapter
	20527	Adapter, 1.0-in. probe rod pin to 1.0-in. NPT pin
	20528	Adapter, 1 25-in. probe rod pin to 1.0-in. NPT pin
	20529	Adapter, 1.5-in. probe rod pin to 1.0-in. NPT pin
	22759	Adapter, 2.0-in. PVC to 1.0-in. NPT Pin
	17558	Adapter, 1.0-in. PVC to 1.0-in. NPT Pin
	19424	Adapter, .75-in. PVC to 17558 Adapter
	17559	Adapter, .5-in. PVC to 17558 Adapter

Table 4.2: Adapters for attaching the outer tube to probe rods and PVC riser pipe.

11. Lower the mechanical bladder pump down the probe rods or well riser. Secure the outer tubing adapter by threading it onto the top probe rod or sliding it over the top of the well riser.

    The mechanical bladder pump is now ready for purging and/or sampling.

Operation of the mechanical bladder pump with a manual actuator is limited to simply raising and lowering the hand grip assembly using a stroke length up to 6 inches (152 mm). This action extends and retracts the pump bladder to push formation fluid to the ground surface through the inner tube of the concentric tubing set. The outer tube is attached to the probe rod string or well riser by adapters and is thus held stationary while the pump is actuated.

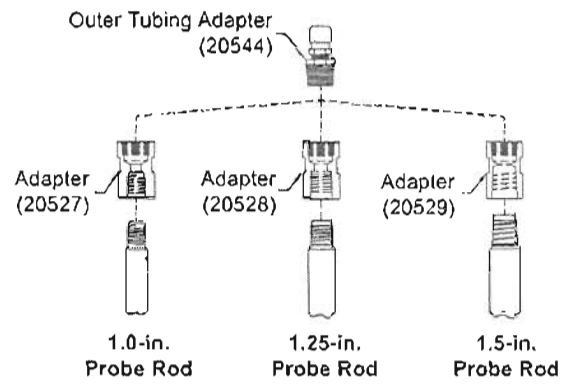

Figure 4.8: Adapters for attaching the outer tube to 1.0-, 1.25-, and 1.5-inch probe rods.

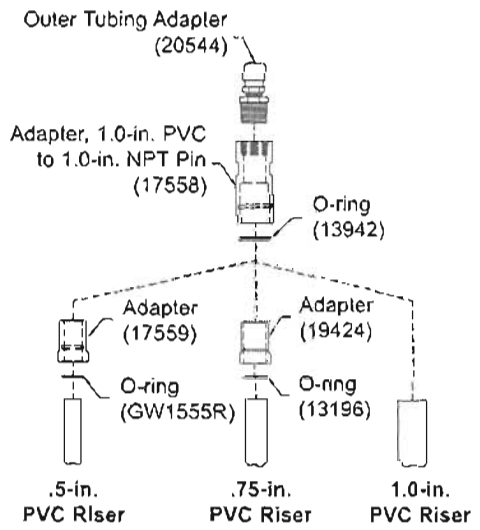

Figure 4.9: Adapters for attaching the outer tube to .5-, .75-, and 1.0-inch PVC riser pipe.

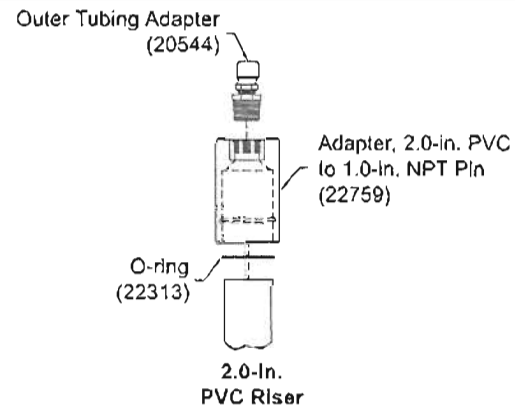

Figure 4.10: Adapters for attaching the outer tube to 2.0-inch PVC riser pipe.

Mechanical Actuator

The third actuator option for the MB470 Mechanical Bladder Pump is a Mechanical Actuator Assembly (MB6000, Figure 4.10). Rather than physically raising and lowering the inner tube to cycle the pump, the operator simply rotates the handle on the side of mechanical actuator. The actuator assembly converts this rotational action to vertical movement of the inner tube which cycles the pump. The operator may also choose to manually raise and lower the inner tube by disconnecting the side handle and utilizing the T-handle at the top of the assembly.

An advantage of the mechanical actuator option is that it requires little physical input to operate the pump. This translates to minimal operator fatigue when purging or sampling from multiple wells during the day.

The mechanical actuator assembly may be installed directly on a probe rod string (Fig. 4.10) or attached to a flush-mount or aboveground well protector using a Well Mount Kit (MB7200) as shown in Figures 4.11 and 4.12. Installation and operation of the mechanical actuator are described below.

1. Determine the depth to which the pump inlet will be installed as measured from the top of the probe rods or well protector with a weighted tape or water level meter.

2. The distance from the pump inlet to the top of the tool string or well protector (from Step 1) may now be marked on the outer tube:

   Obtain an assembled MB470 Mechanical Bladder Pump (Section 4.1) with a concentric tubing set installed as instructed in Section 4.2. Beginning from the pump inlet, measure the appropriate distance along the outer tube and mark it with electrical tape or a suitable marker. The tubing set will be installed such that this mark is aligned with the top of the probe rods or well protector.

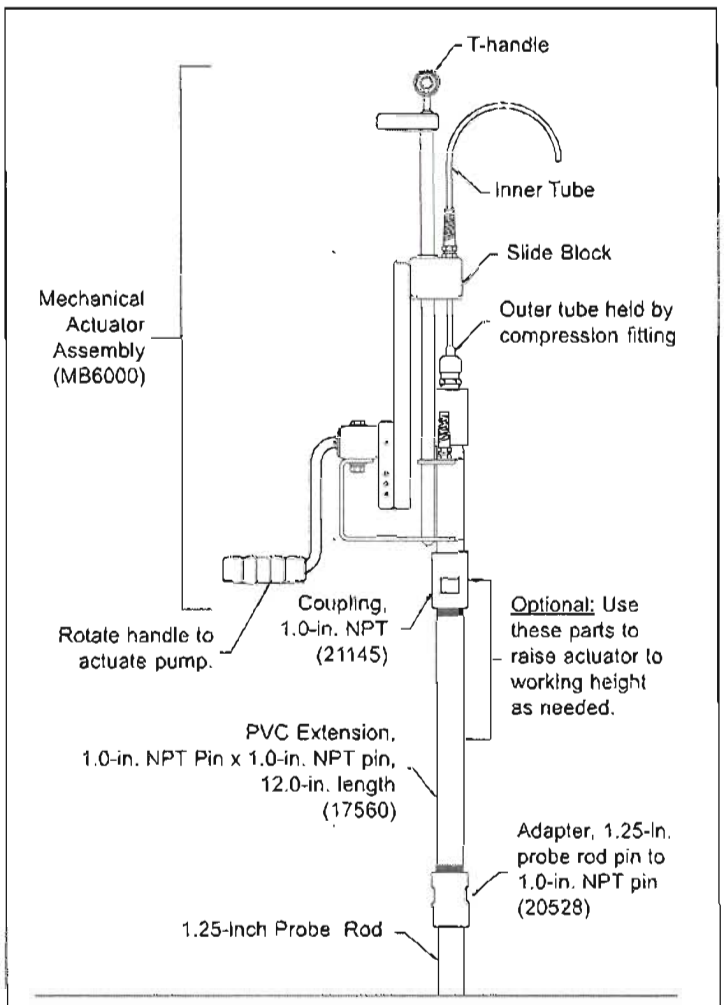

Figure 4.10: Mechanical actuator option for the MB470 Mechanical Bladder Pump (parts shown for attachment to 1.25-inch probe rods).

3. ***For monitoring wells only:*** Install a Well Mount Kit (MB7200, Figure 3.1) on the well protector. The well mount is strapped onto aboveground well protectors as shown in Figure 4.11 and bolted onto flush-mount well protectors as shown in Figure 4.12. Note that the cross adapter is used for flush-mount protectors that utilize three bolts on the cover (Fig. 4.12) or when the well riser is significantly off center in the protector.

4. Lower the pump and concentric tubing set down the probe rod string or through the well mount into the riser pipe. Stop when the mark on the outer tube (from Step 2) is near the top of the probe rods or well protector.

5. ***For probe rods only:*** Referring to Table 4.2, select the appropriate Probe Rod Pin to 1.0-inch NPT Pin Adapter (20527, 20528, or 20529) to attach the actuator to the top probe rod. Thread this adapter (and a 12-inch extension if additional height is needed) into the actuator as shown on the completed assembly in Figure 4.10.

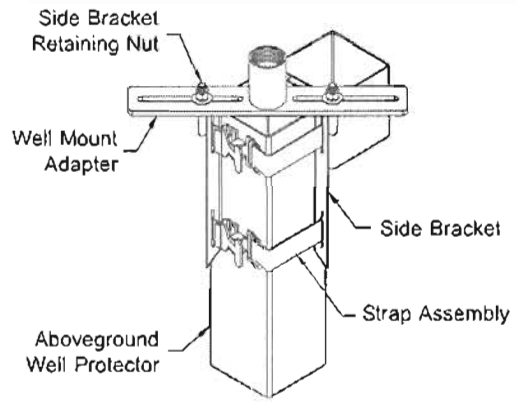

Figure 4.11: Well Mount Kit installed on an aboveground well protector. (Kit may also be used with round protectors.)

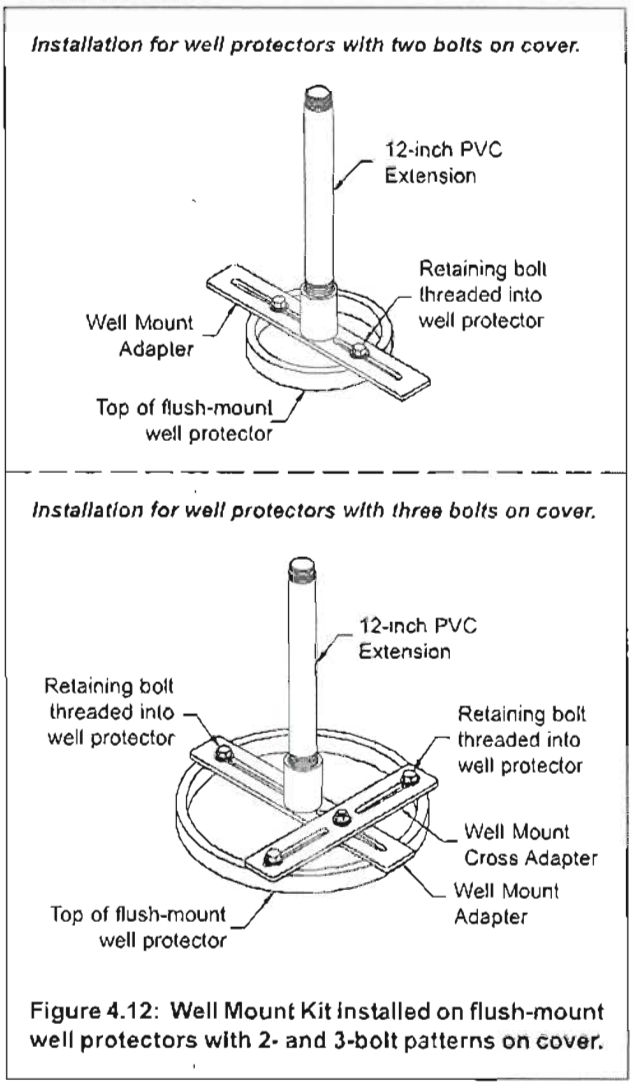

Figure 4.12: Well Mount Kit installed on flush-mount well protectors with 2- and 3-bolt patterns on cover.

6. Insert the top end of the concentric tubing set through the lower end of the mechanical actuator assembly. Feed the tubing set through the actuator and out the compression fitting identified in Figure 4.10.

   For probe rods only: The mark on the outer tube (Step 2) will not be visible once the actuator is installed on the probe rods. To allow for this, position the tubing within the actuator such that the mark will be at the top of the rods when the actuator is installed. Now mark the outer tube at the compression fitting of the actuator assembly for reference later in the installation procedure.

7. Thread the mechanical actuator onto the top probe rod or well mount until all connections are hand tight.

8. Verify the position of the outer tube by observing the mark placed on the tube in Step 2 or 6. Tighten the compression fitting (hand tight) to secure the tubing. Do not overtighten as this may damage the fitting.

9. Carefully cut off the excess outer tube leaving approximately .25 inches (6 mm) above the compression fitting. (Note that the inner tube is not cut at this location).

10. Taking care not to kink the inner tube, insert the inner tube up through the compression fitting on the actuator slide block (see Fig. 4.10 for identification of slide block). It may help to raise the slide block during this step.

    With the slide block fully lowered, gently pull up on the inner tube to remove slack. Do not pull so far that the pump spring is compressed. Tighten the compression fitting to secure the inner tube. Again, do not overtighten as this may damage the plastic fitting.

11. Cut the inner tube leaving it approximately 3 feet (1 m) longer than the outer tube. You may choose to insert the end of the inner tube through the top of the compression fitting on the side of the actuator. This will limit movement of the tube outlet while operating the pump

    The mechanical bladder pump is ready for operation by rotating the side handle of the mechanical actuator or disconnecting the side handle linkage and manually raising and lowering the T-handle.

4.4 Purging and Sampling

The MB470 Mechanical Bladder pump was designed to provide an economical and efficient method to conduct the low flow sampling protocol (Puls and Barcelona 1996, ASTM 2003), Nielsen and Nielsen 2002). The basis of this protocol is that a sampling flow rate of 500 ml/min or less for 2-inch wells (100 to 200 ml/min for smaller diameter direct push wells) generally provides a sample of higher quality that is more representative than sampling at high flow rates (e.g. several liters or gallons per minute). Higher quality samples for volatile organic compounds are obtained because the water being sampled is subjected to less physical and chemical stress so that loss of these analytes does not occur. Additionally, higher quality samples for inorganic analytes (e.g. lead, hexavalent chromium, etc.) are obtained because the low flow sampling method minimizes turbidity that can cause significant bias for these sensitive analytes.

To obtain the most representative samples, the monitoring well or temporary groundwater sampler should be developed before sampling is conducted. Development may consist of simple surging and purging with an inertial pump for temporary samplers depending on the data quality objectives (Geoprobe® 2002). However, more elaborate methods may be required for some monitoring wells (ASTM 2001).

To meet the full requirements of the low flow sampling protocol, field parameters of the pre-sample purge water (temperature, pH, specific conductance, ORP, DO, and turbidity) should be monitored using an in-line flow cell. Once these parameters have stabilized, the samples are then collected in clean, preserved sample containers appropriate for the analytes of concern. Pre-sample purging may be completed in as little as 10 to 20 minutes in adequately developed small-diameter wells with as little as 5 to 10 liters of water generated. In larger diameter wells that have not been adequately developed, a significantly longer purge time and volume may be required.

4.5 Decontaminating the Pump

Decontamination of the pump may be performed in two general ways. For the highest integrity samples the pump should be fully disassembled for thorough decontamination (decon) and the bladder and O-rings replaced. If the pump is being used as a portable pump for sampling multiple locations daily, the pump may be decontaminated while assembled. Review and understand the sampling and data quality objectives for your project before selecting the appropriate decontamination procedure . (For further information on data quality objectives see EPA 1997, or Geoprobe® 2002). The concentric tubing set should be replaced between each sampling location to minimize the potential for cross contamination. If possible, sample from background or low concentration wells to higher concentration wells to minimize the chance for cross contamination.

Disassemble for Decontamination

Simply reverse the procedures described in Section 4.1 to disassemble the pump and concentric tubing set. Place the disassembled pump in a clean beaker or small bucket of water. Use distilled water for highest level of decon. Add Alconox soap (or similar cleaning agent) to the water. Thoroughly clean and brush all inside and outside surfaces. The MBP Cleaning Brush Kit (MB7300) includes four small-diameter brushes selected specifically to clean inside the various pump components. Double rinse all parts with distilled or deionized (DI) water and allow to air dry. Reassemble the pump using a new bladder and O-rings.

Review ASTM Practice D5088 for further guidance and detail on decon procedures. Additional decontamination may be obtained by drying the disassembled pump in a clean drying oven at about 95°C (203°F). This will provide additional assurance that volatile contaminants are removed from pump surfaces.

Decontamination of Assembled Pump

While this method will not provide the assurance of the highest quality samples it may be preferred when lower sample quality is acceptable (For further information on data quality objectives see EPA 1997, or Geoprobe® 2002). When initial site assessments are conducted it is often desirable to obtain many samples at a reasonably modest cost so as to adequately characterize a site. This decon procedure will help reduce the per sample cost while providing acceptable sample quality for many site assessments.

Remove the concentric tubing set from the pump and discard. Submerge the pump in clean soapy water and pump several volumes of water through the pump. Thoroughly wash the exterior of the pump removing all visible dirt or stains. Rinse and transfer the pump to a container of clean tap water or deionized water. Again pump multiple volumes of water through the pump and wash the pump exterior to remove all soap. A second rinse is recommended. Allow the pump to air dry. Again, drying the fully assembled pump in a clean drying oven at about 95°C (203°F) will further remove any volatiles from pump surfaces.

Rinsate Samples

Regularly collect rinsate samples from the pump following decontamination and submit the samples for analysis for the analytes of concern . This will provide another level of quality control and assurance that samples meet the site-specific data quality objectives. Pump clean distilled water through the pump and collect the fluid in an appropriate preserved container. Store, ship and handle rinsate samples in the same manner as field samples.

5.0 REFERENCES

American Society of Testing and Materials (ASTM), 2003. D6771-02 Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations. ASTM, West Conshocken, PA. (www.astm.org)

American Society of Testing and Materials (ASTM), 2001. D-5521 Standard Guide for Development of Ground-Water Monitoring Wells in Granular Aquifers. ASTM, West Conshocken, PA. (www.astm.org)

American Society of Testing and Materials (ASTM), 1999. D-5088 Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites. ASTM, West Conshocken, PA. (www.astm.org)

U.S. Environmental Protection Agency (EPA), 2003. Environmental Technology Verification Report: Geoprobe Inc., Mechanical Bladder Pump Model MB470. Office of Research and Development, Washington, D.C. EPA/600R-03/086. August.

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Parker, Louise V. and Thomas A. Renney. 1998. Sampling Trace-Level Organic Solutes with Polymeric Tubing, Part 2. Dynamic Studies. Groundwater Monitoring & Remediation (GWMR) Vol. XVIII No. 1, pages 148-155. Winter.

Puls, Robert W., and Michael J. Barcelona, 1996. Ground Water Issue: Low-Flow (Minimal Drawdown) Ground Water Sampling Procedures. EPA/540/S-95/504. April.

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APPENDIX K-L

VAPOR INTRUSION INVESTIGATION LETTER WORK PLAN

CONESTOGA-ROVERS
& ASSOCIATES

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December 17, 2010 Reference No. 038443-89

Ms. Karen Cibulskis
Remedial Project Manager
United States Environmental Protection Agency
Region V
77 West Jackson Boulevard
Mail Code SR-6J
Chicago, IL 60604

Dear Ms. Cibulskis:

Re: Vapor Intrusion (VI) Investigation Work Plan (Work Plan)
South Dayton Dump and Landfill Site Moraine, Ohio (Site)

As required under the Dispute Resolution Agreement signed by the Respondents and USEPA on December 10, 2010, this Work Plan presents the proposed approach for a VI Study to investigate sub-slab soil vapor conditions beneath buildings on particular Site parcels and adjacent to the Site. The VI Study will be completed as an interim response action pursuant to Paragraph 37(c) of the Administrative Settlement Agreement and Order on Consent for Remedial Investigation/Feasibility Study (RI/FS) of the Site, Docket No. V-W-06-C-852 (ASAOC). Conestoga-Rovers & Associates (CRA) has prepared this Work Plan on behalf of the Respondents to the ASAOC (Respondents).

The work proposed in this Work Plan will be performed in accordance with the United States Environmental Protection Agency- (USEPA-) approved Field Sampling Plan (FSP), Quality Assurance Project Plan (QAPP), and Site-Specific Health and Safety Plan (HASP), and associated addenda that are submitted as attachments to this Work Plan.

This Work Plan is presented in the following titled sections:

1.0 Background
2.0 VI Study
3.0 Schedule
4.0 Reporting

1.0 BACKGROUND

The Respondents to the ASAOC include Hobart Corporation (Hobart), Kelsey Hayes Company (Kelsey-Hayes), and NCR Corporation (NCR). These three Respondents (the PRP Group) are and have been performing the Work required by the ASAOC under the direction and oversight of the USEPA.

The investigation of the Site has documented elevated concentrations of methane, naphthalene, and volatile organic compounds (VOCs) in landfill gas. There are a number of operating businesses located on the Site, above or immediately adjacent to fill material and in close proximity to the soil gas probe locations where elevated levels of VOCs and methane were detected. By a letter dated October 5, 2010, USEPA had directed Respondents to submit a work plan for a VI Study to address the risks from VI to residents and businesses in buildings on and adjacent to the Site.

VI is the migration of volatile chemicals from the subsurface into overlying buildings. VI is a potential concern at any building, existing or planned, located near soil or groundwater contaminated with toxic chemicals that can volatilize.

Under the December 10, 2010 Dispute Resolution Agreement the Respondents and USEPA agreed that the Respondents will complete the VI Study to assess the potential for methane, VOCs, and naphthalene in soil vapor to result in potential risks to receptors in buildings on and adjacent to the Site.

Specifically, the Dispute Resolution Agreement states:

> [T]*he Respondents shall conduct the VI Study, as required by EPA, pursuant to Paragraph 37 (c) of the ASAOC, as an interim response action. EPA has given the Group a copy of the newly issued EPA Region 5 Vapor Intrusion Guidebook (Guidebook) and the Parties have agreed that the Respondents will prepare their VI Work Plan, which will include Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP) Addenda, in accordance with this new guidance and other relevant guidance (e.g., FSP and QAPP guidance). The Parties agree that the Work Plan will provide for sub-slab sampling, on an expedited schedule of any of the following structures which are of slab-on-grade construction or have basements or enclosed crawl spaces (see highlighted structures on Figure* [1]*, attached, for an illustration of the structures*
> *for which sub-slab sampling is anticipated):*

A. Structures On Site West of Dryden Road:
3 building structures on Lot 5054
3 building structures on Lot 5171
2 building structures on Lot 5172
1 building structure on Lot 5174
1 building structure on Lot 5175, and

B. Structures On Site or Adjacent to Site Along East River Road:
4 building structures on Lot 4610 (Barnett; on-Site)
2 building structures on Lot 3207
1 residence on Lot 3253; and
1 building structure on Lot 3254.

Any additional structures on the Site that are, or may be, occupied will be evaluated to determine the need for VI sampling.

The Parties agree that if any structure on or adjacent to the Site that is or may be occupied has no slab (e.g., dirt or gravel floor) that Respondents will take indoor air samples (see Section 6.6 of Guidebook).

The Parties agree that the Respondents shall submit a Work Plan for the VI Study required by EPA by December 17, 2010. The Parties agree that if identified contaminant concentrations pose more than a 1×10^{-4} cancer risk or a hazard index greater than 1.0 through the VI pathway to current or potential future receptors, or if VI sampling results show an exceedance of 10% of the Lower Explosive Limit, EPA may require actions to mitigate those risks.

The PRP Group has prepared this Work Plan based on requirements of the Dispute Resolution Agreement, previous investigation results and discussions between the PRP Group and USEPA.

2.0 VI STUDY

CRA will complete a sub-slab soil vapor quality investigation beneath the existing on-Site structures and certain structures adjacent to the Site as described in Section 1.0 above. CRA will install and sample the sub-slab soil vapor probes in accordance with CRA's SOPs for installing sub-slab probes and collecting sub-slab soil vapor samples presented in Attachment A, which is an addendum to the FSP.

For any of the structures listed above and any additional structures evaluated that are or may be occupied but do not have a concrete slab floor (e.g., dirt or gravel floor), CRA will collect indoor air samples within the structure. The standard operating procedure (SOP) for the Indoor

Air Sampling is provided in Attachment B (addendum to the FSP). For any location where an indoor air sample is collected, CRA will also install a soil vapor probe screened between 3 and 5 feet below ground surface in accordance with CRA's SOP (Appendix J-F-11 of the FSP) in order to attempt to correlate indoor air concentrations to concentrations of contaminants in soil vapor near the structure. The soil vapor probes will be installed immediately adjacent to the side of the building closest to the most likely source of any soil vapor impacts. CRA will agree on the proposed soil vapor probe locations with USEPA prior to their installation. CRA will collect a soil vapor sample from any newly installed soil vapor probe, and submit the sample(s) for analysis of VOCs by USEPA's TO-15 methodology[1]. In addition, where indoor air samples are collected, CRA will also collect ambient air samples immediately adjacent to the structure as per CRA's SOP. Sub-slab soil vapor and indoor air sampling activities are summarized in Attachment C (addendum to the QAPP).

CRA has prepared this scope of work for the sub-slab soil vapor sampling in accordance with the following vapor intrusion guidance documents:

- Office of Solid Waste and Emergency Response (OSWER) - Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance), November 2002 (USEPA, 2002)
- Interstate Technology Regulatory Council (ITRC) - Vapor Intrusion Pathway: A Practical Guide, January 2007 (ITRC, 2007)
- United States Environmental Protection Agency (USEPA) – Region 5 - Vapor Intrusion Guidebook, October 2010 (USEPA, 2010)

The purpose of the VI Investigation is to collect additional data to determine if compounds are volatilizing into soil vapor beneath the building foundations and floor slabs at concentrations that are sufficiently high that contaminants could potentially migrate into the indoor air of the Site buildings at concentrations that pose an unacceptable risk to building occupants.

A simplified discussion of the DQO steps for the VI investigation is presented below.

Step 1: State the Problem – Soil vapor samples collected from soil gas probes adjacent to three on-Site buildings, and 50 feet from a fourth building, contained VOC concentrations greater than 1×10^{-4} and/or HI=1 industrial risk-based levels. As detailed in the Dispute Resolution Agreement,

[1] Samples will be submitted for USEPA TO-15 GC/MS analysis operated in either select ion monitoring (SIM) or scanning (SCAN) mode, as needed in order to meet required detection limits.

There are a number of operating businesses located on the Site, above or immediately adjacent to fill material and in close proximity to the gas probe locations where elevated levels of VOC and methane were detected.

In addition, there is at least one residential building located in close proximity to soil vapor probe GP09-09, where elevated concentrations of VOCs were detected.

It is not known whether concentrations of contaminants in soil vapor and shallow groundwater pose an unacceptable risk, via the vapor intrusion pathway, to occupants of structures on, or immediately adjacent to, the Site.

Step 2: Identify the goals of the study – Determine whether contaminant concentrations pose more than a 1×10^{-4} cancer risk or a HI greater than 1.0 through the VI pathway to current or potential future receptors. Further, determine whether concentrations of combustible gases within a structure exceed 10 percent of the Lower Explosive Limit (LEL) for methane. Identify buildings where indoor air sampling is required based on the sub-slab sample results.

Step 3: Identify information inputs – Conduct sub-slab soil vapor or, where a structure does not have a concrete slab, indoor air sampling to determine VOC concentrations, through the installation and sampling of sub-slab soil vapor probes and, where appropriate, the collection of indoor air samples.

Step 4: Identify the boundaries of the study – The buildings included in the VI Study are detailed in Section 1.0 above, and presented on Figure 1.

Step 5: Develop the analytic approach – Sub-slab soil vapor samples will be collected from the sub-slab soil vapor probes, following purging in accordance with the FSP. Sub-slab soil vapor and indoor air samples will be submitted for analysis of VOCs in accordance with the requirements of the QAPP and USEPA Method TO-15.

Step 6: Specify Performance or Acceptance Criteria – performance criteria consist of identifying VOC concentrations within existing structures that pose more than a 1×10^{-4} cancer risk or a HI greater than 1 to current or potential future receptors via the vapor intrusion pathway, or an exceedance of 10 percent of the LEL. Additional data quality performance and acceptance criteria are outlined in the QAPP.

Step 7: Develop the plan for obtaining data – see Sections 3.1 to 3.2 below, for detailed procedures proposed in order to obtain the required data.

The sub-slab soil vapor investigation is discussed in further detail below.

2.1 Installation of Sub-Slab Soil Vapor Probes

CRA will assess the potential for vapor intrusion by installing and sampling permanent sub-slab soil vapor probes within the on-Site buildings. The proposed sample locations are presented on Figure 1.

Prior to conducting the sampling, CRA will visually inspect the Lots in question and document the number and type of buildings present on each Lot in order to ensure that all buildings that are or may be occupied are included in the sampling program. Lean-tos, car ports, kennels (unless contained within a larger building), open-sided buildings, etc. will not be included in the sampling program. For buildings where explosive gases might accumulate but exposure times, with respect to specific contaminants would typically be small (i.e., small sheds and outbuildings that do not permit long term exposure), CRA will measure the concentration of explosive gas within the building but will not install a sub-slab soil vapor probe or collect an indoor air sample.

Prior to installing the sub-slab probes, a survey will be conducted of each building, to identify potential preferential pathways for vapor migration under the building. The survey will evaluate the presence of underground utilities, floor slab condition, foundation footings, and vadose zone soil conditions known from nearby monitoring well installations. As building-specific conditions dictate, the probes will be installed in the lowest point of the building, at the approximate middle of the building floor slab. The actual locations will be finalized and documented in the field based on the conditions encountered, the presence of underground utilities, potential preferential vapor migration pathways, and detected groundwater concentrations in the vicinity of each building. The final sub-slab probe locations will be selected with a bias to providing the highest anticipated sub-slab soil vapor concentrations determined based on the weight of the available data collected during the building surveys.

USEPA, 2010 recommends the collection of at least one sample per property and that multiple sub-slab probes be installed in a minimum of 10 percent of the buildings included in the investigation. Therefore, CRA will initially install one sub-slab soil vapor probe per building for eighteen (18) of the twenty (20) buildings included in the investigation. For the remaining two buildings, two sub-slab probes will be installed. These two buildings will be selected based on results of the building survey, the potential presence of multiple preferential exposure pathways, and proximity to elevated groundwater concentrations. In each of the two buildings, one probe will be located at the approximate middle of the building, and the second probe will be located where the greatest degree of variability in sub-slab soil vapor concentrations may be expected based on the weight of the available data collected during the building surveys. If the owners of the residence on Lot 3253 grant permission, and should construction considerations

allow (i.e., underground utilities, floor materials and floor condition), two sub-slab probes will be installed in the residence on Lot 3253, as residents are considered a more sensitive receptor to exposure via the VI pathway.

CRA will complete the sub-slab soil vapor sampling in accordance with CRA's SOPs for collecting sub-slab soil vapor samples (Attachment A, addendum to the FSP). Based on the analytical results of the initial sampling round, CRA will assess the need to install additional sub-slab soil vapor probes to delineate the lateral extent of impact and to identify the maximum sub-slab soil vapor concentrations in the affected building.

As described in detail in CRA's SOP for sub-slab soil vapor probe installation (Attachment A, addendum to the FSP), CRA will use a concrete corer to drill a "shallow" (approximately 1-inch deep) outer hole (approximately 7/8 inches in diameter) that partially penetrates the floor slab. CRA will then use an electric hammer to drill a smaller diameter inner hole (approximately 3/8 inches diameter) into the center of the outer hole, through the floor material and approximately 3 inches into the sub-slab bedding material to create an open cavity.

CRA will clean cuttings from the outer and inner holes using a towel moistened with distilled water or a small portable vacuum cleaner.

To construct the probes, CRA will cut chromatography grade 316 stainless steel or brass tubing (approximately 1/4–inch in diameter) to a length that allows the probe to float within the slab thickness to avoid obstruction of the probe with sub-slab bedding material. CRA will construct the probes prior to drilling to minimize exposure time, or venting, of the sub-slab bedding material through the open hole.

CRA will place the sub-slab soil vapor probe in the hole so that the top of the probe is flush with the top of the floor. The top of the probe will have a recessed stainless steel or brass plug. CRA will push or inject quick drying Portland cement slurry into the annular space between the probe and the outer hole. The cement will be allowed to dry for at least 24 hours prior to sampling.

2.2 Sub-Slab Soil Vapor Probe Sampling

As detailed in the Interstate Technology & Regulatory Council (ITRC) January 2007 document entitled "Vapor Intrusion Pathway: A Practical Guideline":

> *Precipitation can affect vapor intrusion rates and possible soil gas concentrations. Percolation of water through the soil can displace soil gas and lead to a short-term spike in vapor intrusion. The increased soil moisture after a rain event can reduce vapor*

transport through the soil due to reduced effective porosity and permeability. Measurements made during or immediately after a significant rain event (e.g., >1 inch) may not be representative of long-term average conditions.

As per CRA's SOP in Attachment A, sub-slab vapor sampling will not be performed during or within 48 hours of a significant rainfall event (e.g., greater than 0.5 inches of total precipitation).

CRA will collect and submit the sub-slab soil vapor samples for analysis of benzene, toluene, ethylbenzene, and xylenes, along with chlorinated volatile organic compounds (CVOCs) including perchloroethylene (PCE), trichloroethylene (TCE), cis/trans-1,2-dichloroethylene (1,2-DCE), 1,1-dichloroethylene (1,1-DCE), and VC in accordance with the USEPA Toxic Organics-15 (TO-15) parameter list. CRA's SOP for sub-slab vapor probe sampling is described in detail in Attachment A (addendum to the FSP), and is summarized below.

Prior to sampling, CRA will purge the sub-slab soil vapor probes using a personal sampling pump at a flow rate of less than 200 mL/min. This ensures that the sub-slab soil vapor sample is representative of actual vapor concentrations within the sub-slab bedding material. Prior to purging, CRA will complete a vacuum or tightness test on the sampling assembly to test for leaks (details provided below). CRA will purge two to three purge volumes from the probe assembly prior to collecting the samples from each probe using 6-liter Summa®canisters.

The OSWER, ITRC and Region 5 VI guidance documents do not mandate a required minimum number of sampling events to confirm the results. As such, CRA will collect a minimum of two samples from each location and determine the need for additional sampling events based on the initial two sample results. CRA will resample all of the sub-slab soil vapor probe or indoor air sample locations within no less than three months of the collection of the initial sample to account for seasonal changes. Locations selected for sampling on a second occasion will be sampled at least once during the winter when the surrounding ground is frozen and vapor intrusion is expected to be highest.

Where contaminants are detected in a sub-slab soil vapor or indoor air sample at concentrations that represent an excess lifetime cancer risk above 1×10^{-4} or a non-cancer HI greater than 1, CRA will collect a confirmatory sample as soon as reasonably practicable following receipt of the sample results. Where contaminants are detected in both the original and confirmatory sub-slab soil vapor samples at concentrations that exceed an excess lifetime cancer risk above 1×10^{-4} or a non-cancer HI greater than 1, CRA will collect two indoor air samples (in two discrete sampling events) to determine whether the contaminants detected in the sub-slab samples are migrating to indoor air at concentrations that pose an unacceptable risk to receptors.. The indoor air samples will be collected following the procedures described above and in accordance with the relevant requirements of the FSP and QAPP.

2.2.1 Leak Testing

Prior to purging, CRA will complete a vacuum test on the sampling assembly as the first of two leak-testing steps. During the first leak-testing step, CRA will open the valve to the personal sampling pump leaving the valves to the Summa™ canister and the soil gas probe closed. CRA will then operate the pump to ensure that no ambient air enters the sampling assembly (i.e., the pump should create a negative pressure within the sampling assembly).

During the second leak-testing step, CRA will release a tracer compound to the ground surface immediately around the sub-slab probe surface casing. The tracer test will test for ambient air leakage through the probe assembly. The tracer compound is either monitored for in the field using a meter connected in-line to sampling assembly (e.g., helium), or is included as an analyte in the laboratory analysis of the soil gas samples (e.g., isopropanol). CRA will complete leak testing during sample collection by injecting helium into a shroud covering the sub-slab probe, and monitoring for the presence of helium in the sampling line both before and after sample collection.

Attachment A (addendum to the FSP) details the protocol for leak testing.

2.2.2 QA/QC

For QA/QC purposes, CRA will submit one field duplicate for every 10 samples submitted. Based on the total expected sub-slab soil vapor samples during the initial sampling round, CRA will submit two field duplicate samples. CRA will also submit one trip blank sample for analysis to assess the sample handling procedures, and one background outdoor air sample per day to assess the background concentrations at the time of sampling. Where sampling occurs in more than one area of the Site on a single day, CRA will collect one background outdoor air sample from each area to ensure that local-scale ambient air concentrations of contaminants are characterized. All Summa canisters used in the sampling program will be individually certified by the laboratory to ensure that they are free of contamination prior to collection of the samples. Results of this certification will be included in the VI Investigation Report.

3.0 SCHEDULE

Field work will begin within thirty days of receipt of USEPA approval of the VI Investigation Work Plan, dependant on subcontractor availability, and obtaining access to the various private properties, businesses and residences. Follow-up sampling will be completed within 90 to 120 days of the original sampling event.

4.0 REPORTING

CRA will post the validated analytical results to the South Dayton Dump and Landfill file transfer protocol (ftp) site immediately upon completion of validation. CRA will notify USEPA immediately of any analytical results that demonstrate a potential excess lifetime cancer risk above 1×10^{-4} or a non-cancer HI greater than 1.

The draft VI Investigation Report will be submitted to USEPA within thirty days of receipt of the final laboratory data report from the second sampling event. The draft VI Investigation Report will provide a summary of results from the sub-slab soil vapor and indoor sampling and recommendations for further sampling or remedial actions required to address any unacceptable risks to on- or off-Site receptors. The VI Report will be finalized following receipt of comments from USEPA. Monthly progress reports submitted to USEPA during the investigative work will include the information required for monthly progress reports in the RI/FS SOW.

Should you have any questions on the above, please do not hesitate to contact us.

Yours truly,

CONESTOGA-ROVERS & ASSOCIATES

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VC/ca/98
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Wray Blattner, Thompson Hine
Ken Brown, ITW
Kelly Smith, Terran
Tim Hoffman, Dinsmore & Shohl
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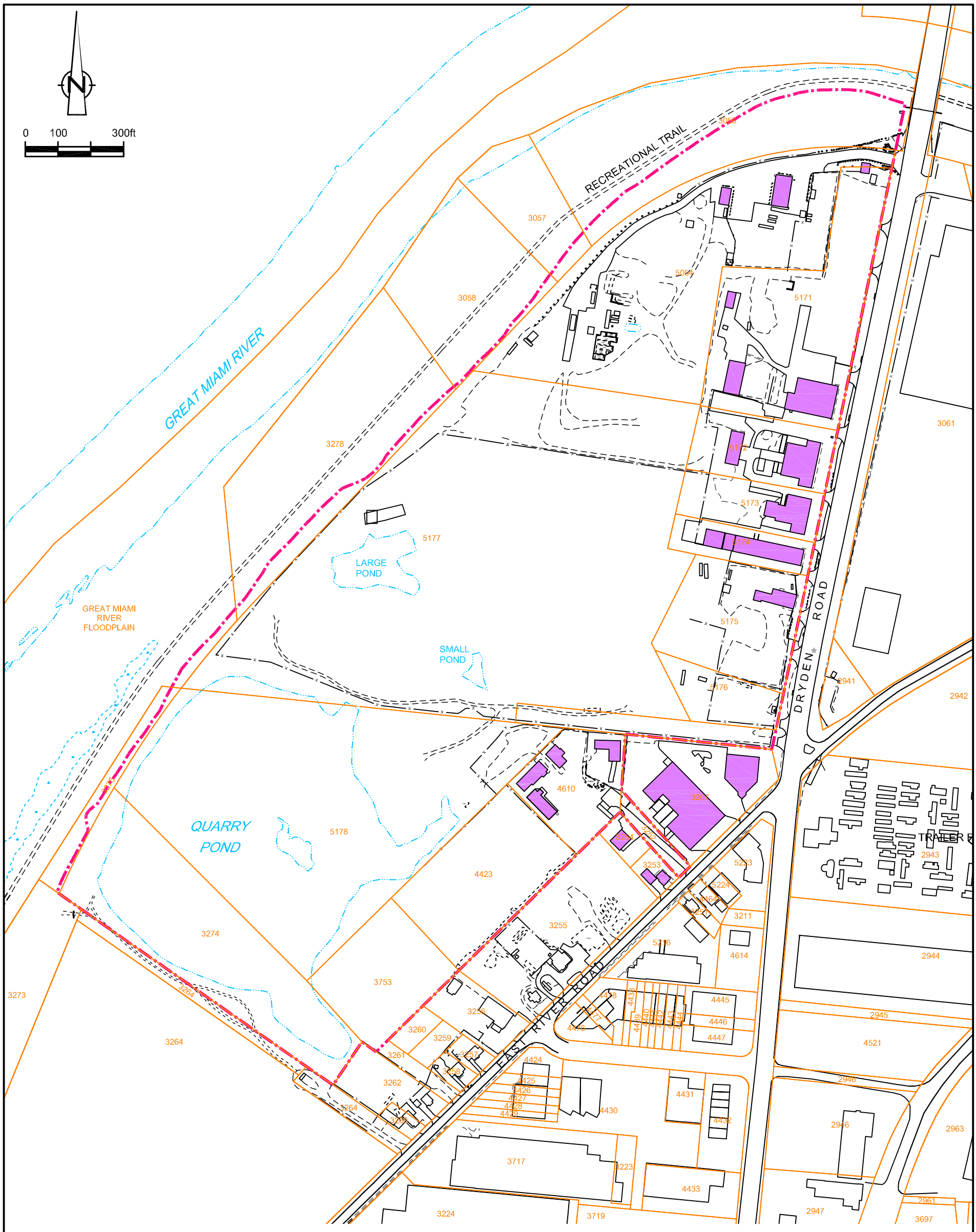
Edward Gallagher, NCR
Karen Mignone, Verrill Dana
Adam Loney, CRA
Jim Campbell, EMI
Chris Athmer, Terran
Bryan Heath, NCR



LEGEND

- APPROXIMATE SITE BOUNDARY
- EDGE OF WATER
- PARCEL BOUNDARY
- SUB SLAB SAMPLING LOCATION

figure 1

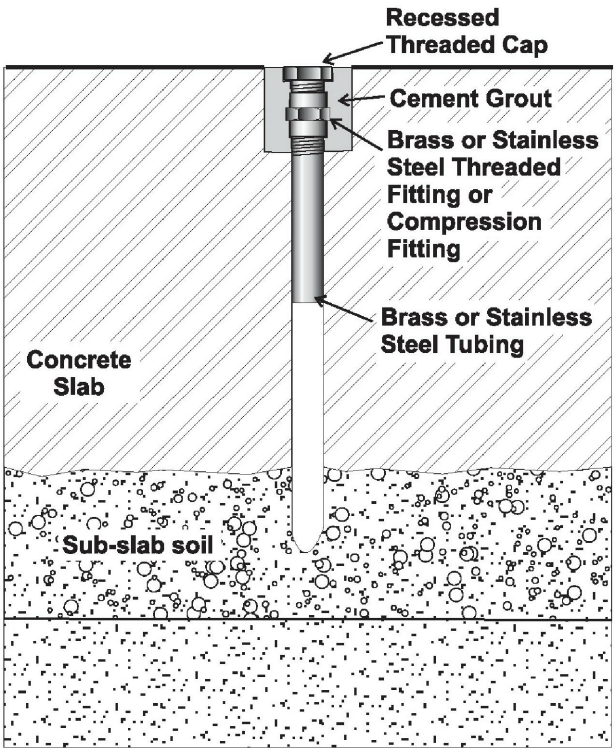
SUB SLAB SAMPLING LOCATIONS
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

SOURCES:
THE PAYNE FIRM, INC., PROJECT 0279.44.05, FIGURE 1, DATED 9/12/05;
TETRA TECH EM INC., PROJECT L0312006-SOUTH DAYTON DUMP, FIGURE 2, SITE LAYOUT, 05/25/2004;
CITY OF MORAINE.
ABRAMS AERIAL SURVEY INC. PROJECT 38443, AASI 29610, 04/02/2008

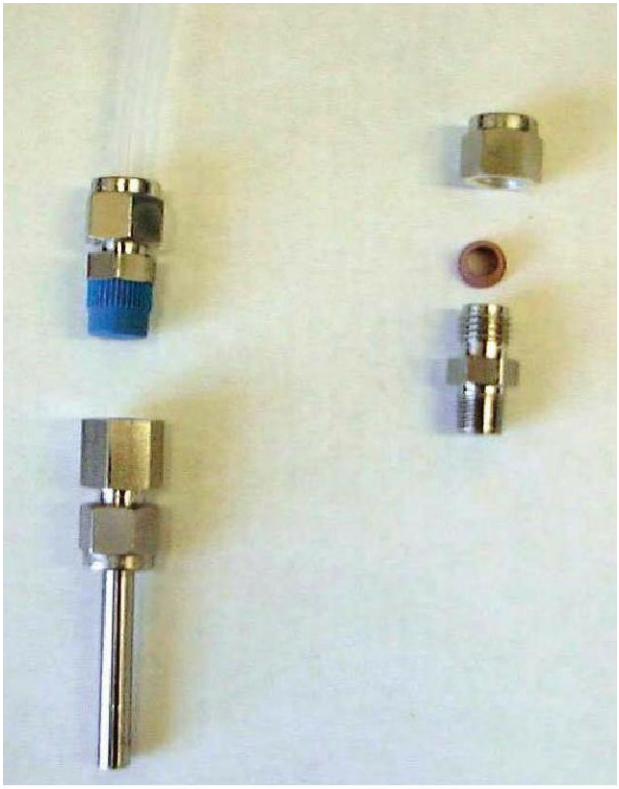
38443-89(CIBU098)GN-WA001 DEC 17/2010

ATTACHMENT A

STANDARD OPERATING PROCEDURE
FOR SUB-SLAB SOIL GAS PROBES



SCHEMATIC OF TYPICAL SUB-SLAB SOIL GAS PROBE



FITTINGS USED FOR SUB-SLAB SOIL GAS PROBE ASSEMBLY

figure A.1

SOURCE: U.S. EPA (2006)

TYPICAL SUB-SLAB SOIL GAS COMPLETION DETAIL
SOUTH DAYTON DUMP AND LANDFILL SITE
Moraine, Ohio

38443-89(CIBU098)GN-WA002 DEC 17/2010

STANDARD OPERATING PROCEDURE FOR
SUB-SLAB SOIL GAS PROBES

1.0 PRIOR PLANNING AND PREPARATION

Prior to installing a sub-slab gas probe:

1. Review the Work Plan and HASP with the Project Coordinator. Understand the existing site geologic/hydrogeologic conditions such as the type of soil, level of water table or perched groundwater table, and properties of refuse (if installing a probe in a landfill) such as depth, leachate levels or perched leachate levels. Know the seasonally high and low water table and leachate elevations, and know if perched conditions exist.
2. Assemble all required equipment, materials, log books, and forms.
3. Coordinate with a drilling/coring contractor (if one is retained) to ensure the work can be completed and to provide them with all relevant information to complete the job prior to arriving on site.
4. Obtain information on the probes to be installed to ensure a complete understanding of the task to be performed. Required information for installation includes knowing the type of gas probe construction materials that are to be used, including knowing the diameter of the probe, depth of probe (length of riser), type and amount of packing material, type of probe material, and planned location for each probe. Also determine if multilevel probes are required.
5. Determine the type of analyses that are required from the probes after installation, and the type of gas monitoring that is required during the drilling and installation of the probe.
6. Arrange access to the site, especially if the property owner is not our client. Obtain all necessary keys. Also consider site conditions (e.g., is snow removal required?).
7. Determine excess soil or refuse disposal procedures before commencing drilling/coring activities.
8. Determine drilling or property access notification requirements with the Project Coordinator. Notify the client, landowner, and appropriate regulatory agencies and complete utility clearance activities in accordance with the FSP.
9. Understand and review the potential health and safety hazards associated with the task and with the site.

These considerations should have been incorporated during development of the Work Plan and should be discussed with the Project Coordinator.

2.0 EQUIPMENT DECONTAMINATION

Prior to use between gas probe locations, drilling and sampling equipment must be decontaminated in accordance with the Work Plan, the Quality Assurance Project Plan (QAPP), or the methods presented in the following section.

The minimal procedures for decontamination of drilling or excavating equipment are:

1. Hot water and detergent wash (brushing as necessary to remove particulate matter).
2. Potable, hot water rinse.

Cover clean equipment with clean plastic sheeting to prevent contact with foreign materials.

On environmental sites, soil sampling equipment (e.g., split-spoons, trowels, spoons, shovels, and bowls) is typically cleaned as follows:

1. Wash with clean potable water and laboratory detergent, using a brush as necessary to remove particulates.
2. Rinse with potable water.
3. Rinse with deionized water.
4. Air dry for as long as possible.

3.0 INSTALLATION PROCEDURES – SUB-SLAB GAS PROBES

Sub-slab soil gas probes allow for collection of sub-slab soil gas samples from directly beneath the slab of a building. Note that sub-slab soil gas probes are not recommended when groundwater is present directly below the slab, as drilling through the slab could allow groundwater to enter the building. A summary of the steps involved in the installation of sub-slab soil gas probes is presented below:

1. Prior to drilling holes into the building floor, the location of utilities coming into the building (e.g., gas, electrical, water, and sewer lines, etc.) will be identified. Avoid installing sub-slab soil gas probes near where utilities penetrate the slab as these may be entry points for downward ambient air migration through the slab during sub-slab soil gas sampling.
2. A rotary hammer drill or equivalent equipment will be used to drill a "shallow" [approximately 1-inch (2.5-cm) deep] outer hole [approximately 7/8 inches (2.2 cm) in diameter] that partially penetrates the floor slab. Cuttings may be removed using a towel moistened with distilled water or small portable vacuum cleaner.
3. The rotary hammer drill or equivalent equipment will be used to drill a smaller diameter inner hole, within the center of the outer hole, approximately 3/8 inch (9.5 mm) in diameter through the floor material and approximately 3 inches (7.6 cm) into the sub-slab bedding material to create an open cavity. The outer hole will be cleaned with a towel moistened with distilled water.
4. Chromatography grade 316 stainless steel or brass tubing will be cut to a length that allows the probe to float within the slab thickness to avoid obstruction of the probe with sub-slab bedding material. The tubing will be approximately ¼ inch (6.4 mm) in diameter. Where necessary, the compression fittings will be stainless steel or brass (approximately ¼ inch O.D. and 1/8-inch NPT) Swagelok® female thread connectors. Whenever possible, the probes will

be constructed prior to drilling to minimize exposure time, or venting, of the sub-slab bedding material through the open hole.

5. The sub-slab soil gas probe will be placed in the holes so that the top of the probe is flush with the top of the floor. The top of the probe will have a recessed stainless steel or brass plug. A quick-drying, Portland cement slurry will be injected or pushed into the annular space between the probe and the outer hole. The cement will be allowed to dry for at least 24 hours prior to sampling.

3.1 INSTALLATION DOCUMENTATION

Details of each sub-slab soil gas probe installation should be recorded on CRA's standard Stratigraphic Log Overburden, or recorded within a standard CRA field book. The Well Instrumentation Log is provided for recording the overburden well instrumentation details, and can be used for sub-slab soil vapor probe installations. This figure must note:

- borehole depth;
- slab thickness;
- probe perforation intervals;
- plug intervals;
- surface cap detail;
- sub-slab soil gas probe material;
- sub-slab soil gas probe instrumentation (i.e., probe length);
- sub-slab soil gas probe diameter;
- cement slurry seal detail;
- stickup/flush-mount detail; and
- date installed.

Each sub-slab soil gas probe installed must have accurate field ties to the center of the sub-slab soil gas probe from three adjacent permanent features of the structure within which the probe is installed, each located in a different direction from the installation.

Each sub-slab soil gas probe must be permanently marked to identify the sub-slab soil gas probe number designation.

4.0 RESPIRATORY PROTECTION

The HASP must be followed with regard to respiratory protection.

5.0 FOLLOW-UP ACTIVITIES

Once the sub-slab soil gas probe(s) have been completed, the following activities need to be done:

1. Conduct initial monitoring round of gas probes.
2. All logs will be submitted to CRA's hydrogeology department who will be responsible for the generation of the final well log.
3. Arrange surveyor to obtain accurate horizontal and vertical control.
4. Gas probe/boring locations will be accurately plotted on the site plan, since boring locations may change in the field due to utility interferences or other conditions.
5. Tabulate sub-slab gas probe details.
6. A summary write-up on field activities including, but not necessarily limited to such items as drilling method(s), construction material, etc.
7. Field book will be kept at the appropriate CRA office.

6.0 FIELD INSTRUMENTATION CALIBRATION

Sampling or monitoring equipment used in the sub-slab soil gas and outdoor air sampling program to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specification and requirements. Field calibration of the personal sampling pump and PID meter will be carried out prior to sampling activities.

The vacuum gauge used to measure canister vacuum will be calibrated and provided by the laboratory. The vacuum gauge will be returned to the laboratory for the laboratory to obtained vacuum measurements prior to sample analysis (checking canister integrity was maintained during shipment). Using a common vacuum gauge will avoid variations in vacuum measurements that can arise due to using different vacuum gauges.

7.0 SUB-SLAB SOIL GAS SAMPLING PROTOCOL

The following sections describe the protocol for sub-slab soil gas sampling from permanent sub-slab soil gas probes. For evaluating vapor intrusion, permanent sub-slab soil gas probes are preferable to allow for multiple sub-slab soil gas sampling events. More than one sub-slab soil gas sampling event is often required when assessing vapor intrusion to address seasonal variations and temporal variability commonly observed in sub-slab soil gas concentrations.

Sub-slab soil gas sampling should commence a minimum of 24 hours following installation of the sub-slab soil gas probes, to allow time for disturbances created by drilling to dissipate and allow the formation to return to an equilibrium condition. In fine-grained soil conditions, consideration should be given to allowing a greater amount of time for equilibrium conditions to become re-established (e.g., 72 hours). Sub-slab soil gas sampling will not be performed during or within 48 hours of a

significant rainfall event [e.g., >0.5 inches after Cal EPA (2003)]. This will avoid the potential that increased moisture content in the unsaturated zone soil could temporarily dampen sub-slab soil gas concentrations, or possibly prevent sub-slab soil gas sample collection (i.e., such as in cases where the sub-slab soil gas probe screened interval could become temporarily saturated due to the passing infiltration front). In fine-grained soil conditions, consideration should be given to allowing a greater amount of time for rainfall events to dissipate. The potential influence of rainfall events on sub-slab soil gas concentrations is less of concern in cases where the sub-slab soil gas probes are located beneath impervious ground cover (e.g., pavement or building foundation).

A summary of the steps involved in sub-slab soil gas sampling is presented below:

i) Sub-slab soil gas samples for assessing the vapor intrusion pathway will be collected using certified clean Summa™ canisters. Only canisters certified clean at the 100 percent level can be used for sub-slab soil gas sampling activities (i.e., pre-cleaned at the laboratory in accordance with U.S. EPA's TO-15 method and documentation of the cleaning activities will be provided by the laboratory). Summa™ canisters typically come in 1-, 1.7-, and 6-liter capacities, depending upon laboratory availability. Consideration should be given to using smaller capacity canisters to reduce sample volume and increase confidence that the sub-slab soil gas sample is drawn from the formation immediately surrounding the probe screen during sampling. Larger volume samples can promote drawing ambient air down the annulus of the sub-slab soil gas probe which can dilute the sub-slab soil gas sample. The use of the smaller canister sizes becomes more critical in fine-grained soil conditions where the formation may not give up significant sub-slab soil gas volumes (in this case, ambient air infiltration down the sub-slab soil gas probe annulus can be more problematic).

ii) The Summa™ canisters will be fitted with a laboratory calibrated critical orifice flow regulation device sized to restrict the maximum sub-slab soil gas sample collection flow rate to approximately 100 milliliters per minute (mL/min), which corresponds to the lower end of the maximum sub-slab soil gas sampling flow rate recommended by Cal EPA (2003) of 100 to 200 mL/min. The 100 mL/min maximum flow rate translates to sample collection times of 10, 17, or 60 minutes, respectively, for of 1-, 1.7-, or 6-liter canister capacities. A maximum flow rate of 100 mL/min is recommended to limit VOC stripping from soil, prevent the short-circuiting of ambient air from ground surface down the sub-slab soil gas probe annulus that would dilute the sub-slab soil gas sample. A maximum flow rate of 100 mL/min increases confidence that the sub-slab soil gas sample is drawn from immediately surrounding the screened interval.

iii) A vacuum gauge will be supplied by the laboratory and used during sample collection to measure the initial canister vacuum, canister vacuum during sample collection, and residual canister vacuum at the end of sample collection. The vacuum gauge will be returned to the laboratory and used by the laboratory to measure the residual canister vacuum upon receipt of the canisters by the laboratory. Using the same vacuum gauge throughout the entire sampling process will eliminate discrepancies between vacuum measurements that can arise from using different gauges with a potentially different sensitivity and/or calibration.

iv) The canister will be connected to the sub-slab soil gas probe valve at the surface casing using the sampling assembly that is depicted on Figure 15.5. The sampling assembly is connected using short lengths [e.g., 1-foot (0.3 m)] 1/4-inch (6.4 mm) or 3/8-inch (9.5 mm) diameter tubing (the tubing material will be Teflon® or nylon) and air-tight stainless steel or brass tee-connectors and tee-valves

(e.g., Swagelok® type). The canister will be connected to the sub-slab soil gas probe along with a vacuum gauge and a personal sampling pump, all in series, using tee-connectors or tee-valves (in the order of sub-slab soil gas probe, vacuum gauge, pump, and canister). A tee-valve will be used to connect the pump, which will allow the pump to be isolated from the sampling assembly during sample collection. Fresh tubing will be used for each sample.

v) Prior to collecting a sub-slab soil gas sample, the stagnant air in the sampling assembly tubes and sub-slab soil gas probe casing/sand pack must be removed. The sub-slab soil gas probes will be purged prior to sampling using the personal sampling pump at a flow rate of less than 200 mL/min. This ensures that the collected sub-slab soil gas sample is representative of actual sub-slab soil gas concentrations within the formation. Measurements of the lengths and inner diameters of the above-ground sampling assembly and below-ground gas probe casing, screen, and sand pack should be used to calculate the "purge volume" (the purge volume will consider the pore volume of the sand pack assuming a 30 percent sand pack porosity). Prior to sample collection, two to three purge volumes should be drawn from the probe/sample assembly, unless otherwise required by the applicable regulatory guidance. The purge data (calculated purge volume, purging rate, and duration of purging) should be recorded in the field logbook.

vi) Prior to purging, a vacuum, or tightness, test will be conducted on the sampling assembly as the first of two leak-testing steps, as described further in Section 15.2.4. Briefly, this first leak-testing step (the vacuum test) will consist of opening the valve to the personal sampling pump leaving the valves to the Summa™ canister and the sub-slab soil gas probe closed. The pump will then be operated to ensure that it draws no air from the sampling assembly (i.e., creates a negative pressure, or vacuum within the sampling assembly), thus establishing that all assembly connections are air-tight. Further details of the vacuum test are described below.

vii) Prior to purging, and following the vacuum test, the set-up for the second of the two leak-testing steps will be conducted. The second leak-testing step is the tracer compound step. A tracer compound is released at ground surface immediately around the sub-slab soil gas probe surface casing. The tracer test is used to test for ambient air leakage down the annulus of the sub-slab soil gas probe and into the sub-slab soil gas sample. The tracer compound is either monitored for in the field using a meter connected in-line to sampling assembly (e.g., helium), or is included as an analyte in the laboratory analysis of the sub-slab soil gas samples (e.g., isopropanol). The set-up requirements of the tracer compound leak-testing step are described below.

viii) Following the vacuum test, and the set-up for the tracer compound leak-testing step, the sub-slab soil gas probe purging will commence by opening the valve to the sub-slab soil gas probe and activating the personal sampling pump (and leaving closed the valve to the Summa™ canister). At the start and the end of the purging period, the total concentration of volatile organic vapors of the personnel sampling pump exhaust gas will be monitored using a portable photoionization detector (PID) meter. The PID meter will be connected in series after the personal sampling pump. Since typical PID instrument flow rates vary from approximately 300 mL/min to 500 mL/min (depending on the manufacturer and model), drawing a sample into the PID meter through the personal sampling pump likely will increase the purging flow rate temporarily until a reading from the PID meter is obtained. PID readings will be recorded and entered in the field logbook and chain of custody form. The PID readings will provide the laboratory with an indication of whether a sample could require dilution before analysis.

ix) Following purging, the valve to the personal sampling pump will be closed, and the valves to the sub-slab soil gas probe and Summa™ canister will be opened to draw the sub-slab soil gas sample into the canister concurrent with continuing to apply the leak-testing tracer compound. The vacuum gauge reading will be recorded during sample collection. Should the vacuum gauge reading remain elevated above 10-inches mercury (Hg) for more than 30 minutes, this will be taken to indicate that the initial vacuum in the canister has not sufficiently dissipated, and that the soil screened by the sub-slab soil gas probe does not produce sufficient sub-slab soil gas to permit sample collection.

x) To ensure some residual vacuum in each canister following sample collection, the canister vacuum will be recorded at approximately 80 percent through the expected sample collection duration. With a 100 mL/min maximum flow rate, the expected sample collection duration would be 10, 17, or 60 minutes, respectively, for canister capacities of 1, 1.7, or 6 liters. A maximum residual vacuum of 10-inches Hg is allowed. A canister residual vacuum above this value will require continued sampling until vacuum reading is below this threshold, unless the vacuum remains above 10-inches Hg for more than 30 minutes, as described above. A minimum 1-inch Hg residual vacuum will be required for the sample to be considered valid, or the sampling will be repeated using a fresh Summa™ canister. Once the vacuum is measured, the safety cap will be securely tightened on the inlet of the Summa™ canister prior to shipment to the laboratory under chain of custody procedures.

xi) The vacuum gauge provided by laboratory will be returned with the canister samples to check residual vacuum in the laboratory prior to sample analysis and recorded on the analytical data report. This check will ensure sample integrity prior to laboratory analysis, and that the canister has not become compromised during shipment to the laboratory.

xii) If the critical orifice flow regulation devices (provided by the laboratory) and sampling assembly fittings/valves are to be re-used during sampling, they will be cleaned in accordance with laboratory requirements by purging with zero air (provided by laboratory) for minimum 45 seconds at minimum 75 psi.

xiii) The canisters will be labeled noting the unique sample designation number, date, time, and sampler's initials. A bound field logbook will be maintained to record all sub-slab soil gas sampling data.

xiv) The canisters will be listed on the chain-of-custody in order of suspected highest to lowest impact, as evidenced by the recorded PID readings. Indicate on the chain-of-custody for the laboratory to analyze the canisters in order from the lowest to highest PID reading.

The sub-slab soil gas samples will be analyzed for VOCs by the project laboratory using U.S. EPA's TO-15 gas chromatograph/mass spectrometer (GC/MS) methodology, with the mass spectrometer (MS) run in full scan mode. Quality control/quality assurance (QA/QC) measures implemented during the sub-slab soil gas sampling event will include the two-step leak testing procedure (see Section 15.2.4), maintaining a minimum residual vacuum in the Summa™ canisters following sample collection, collection of one duplicate per sampling event or from at least 10 percent of the samples obtained, and collection of an ambient air sample.

8.0 SUB-SLAB SOIL GAS PROBE LEAK TESTING

The use of leak testing is recommended as a quality control check to ensure ambient air has not leaked into the sub-slab soil gas probe or sampling assembly, which may affect (i.e., dilute) the analytical results. Contaminants in ambient air can also enter the sampling system and be detected in a sample from a non-contaminated sampling probe resulting in a "false positive" result. The leak testing will be conducted in the following two steps:

- Step 1 - Vacuum Test: used to ensure that the tubing and fittings/valves that make up the sampling assembly are air tight; and
- Step 2 - Tracer Test: used to ensure that ambient air during sub-slab soil gas sample collection is not drawn down the sub-slab soil gas probe annulus through an incomplete seal between the formation and the sub-slab soil gas probe casing.

The vacuum test and tracer test are detailed below.

Step 1 - Vacuum Test

- The sampling assembly will be connected to the sub-slab soil gas probe valve at the surface casing. Once connected, the sampling assembly will consist of the sub-slab soil gas probe, the vacuum gauge supplied by the laboratory, personal sampling pump, and Summa™ canister, all connected in series (i.e., in the order of sub-slab soil gas probe, vacuum gauge, pump, and canister), using tee-connectors or tee-valves.
- The personal sampling pump will be used to conduct the vacuum test. The vacuum test will consist of opening the valve to the personal sampling pump while leaving closed the valves to the Summa™ canister and the sub-slab soil gas probe. The pump will then be operated to ensure that it draws no air from the sampling assembly (i.e., creates a negative pressure, or vacuum within the sampling assembly), thus establishing that all assembly connections are air-tight. The sampling pump low-flow detect switch will likely activate within 10 to 15 seconds, turning the pump off. A negative pressure, or vacuum, should be established within the sampling assembly, and should be sustained for at least 1 minute.
- If the pump is capable of drawing flow, or if the vacuum is not sustained for at least 1 minute, all fittings and tubing will be checked for tightness (or replaced) and the vacuum test will be repeated.
- The reading from the vacuum gauge pressure will be recorded in field logbook to demonstrate that the pump is able to create a vacuum within the sampling assembly (it will also be noted whether the low-flow detect switch on the pump was activated), and that the vacuum is sustained for at least 1 minute.

Step 2 - Tracer Test

A tracer compound is released at ground surface immediately around the sub-slab soil gas probe surface casing and is used to test for ambient air leakage down the annulus of the sub-slab soil gas probe and into

the sub-slab soil gas sample. Two options are described below for the tracer test where either isopropanol (Option A) or helium (Option B) is used as the tracer compound.

Option A - Isopropanol

- For Option A, isopropanol is used as the tracer compound. It is included as an analyte in U.S. EPA's TO-15 method, it is readily available (i.e., as isopropyl rubbing alcohol), and it is safe to use.
- Approximately 1 teaspoon (approximately 4 mL) of isopropanol (rubbing alcohol) will be mixed in 1 gallon of de-ionized water to create an approximate 1/1,000 solution.
- Paper towels soaked in the dilute solution of isopropanol will be wrapped around the sub-slab soil gas probe surface casing and ground surface immediately surrounding the surface casing. Sub-slab soil gas probe surface casing then will be covered over using clear plastic sheeting that will be sealed to the ground surface. As the ground surface finish permits, sealing the plastic sheeting to ground surface will be accomplished using tape or by weighting the edges of the plastic sheeting with dry bentonite.
- Immediately before conducting the sub-slab soil gas probe purging, remove the paper towels from the solution wringing out the towels so they are very damp, but not dripping, before placed them around the vapor probe and sealing them in place using the plastic sheeting.
- The isopropanol solution will be kept fresh, with new solution being made every hour. The solution will be mixed at a central location away from the sampling activities. The isopropanol will be kept tightly capped and kept away from all sampling equipment. The solution will be kept away from the sampling assembly until immediately before sample collection begins. Sampling personnel will wear latex gloves while handling the solution and soaked paper towels, and will remove the gloves while working with the sampling assembly.
- Soil samples with laboratory analytical results for isopropanol that are greater than 10 percent of the starting concentration of isopropanol in the vapors emitted from dilute isopropanol solution will not be considered reliable and representative of sub-slab soil gas concentrations within the formation (ITRC, 2007). The starting concentration will be calculated based on the concentration of isopropanol in the dilute solution, the vapor pressure of isopropanol, and Henry's law.
- A disadvantage in using isopropanol as the tracer compound is that it will not be known whether a significant leak occurred until after the cost of analyzing the sample has been spent. Elevated levels of isopropanol can also interfere with laboratory analytical method detection limits.

Option B - Helium

- The presence of helium within the sampling assembly will be monitored during purging and sub-slab soil gas sample collection using a helium meter installed in-line with the sampling assembly just before the personal sampling pump.
- Helium is readily available at a variety of retail businesses, is safe to use, and does not interfere with laboratory analytical method detection limits.

- A containment unit is constructed to cover the sub-slab soil gas probe surface casing. The containment unit will consist of an over-turned plastic pail set into a ring of dry bentonite to create a seal between the ground surface and the rim of the pail. The pail can be set directly on top of the sampling assembly tubing connected to the sub-slab soil gas probe, which when pressed into the dry bentonite, should create a sufficient seal around the tubing. The pail will have two holes: one to allow for the introduction of helium; and the other to allow for air trapped inside the pail to escape while introducing the helium. The second hole will also allow insertion of the helium meter to measure the helium content within the pail.
- Prior to sub-slab soil gas probe purging, helium will be introduced into the containment unit to obtain a minimum 50 percent helium content level. The helium content within the containment unit will be confirmed using the helium meter and recorded in the field logbook. Helium will continue to be introduced to the containment unit during sub-slab soil gas probe purging and sampling, but care will be taken not to increased the pressure within the containment unit beyond that of atmospheric pressure.
- During sub-slab soil gas probe purging and sampling, the helium meter will be connected in-line with the sampling assembly. In the event that the helium meter measures a helium content with the sampling assembly of greater than 10 percent of the source concentration (i.e., 10 percent of the helium content measured within the containment unit), the sub-slab soil gas probe will be judged to permit significant leakage such that the collected sub-slab soil gas sample will not be considered reliable and representative of sub-slab soil gas concentrations within the formation (ITRC, 2007).
- An advantage of using helium as the tracer compound is that a significant leak can be detected in the field and the cost of analyzing the Summa™ canister can be avoided.

REFERENCES

Cal EPA, 2003. Advisory – Active Sub-slab soil gas Investigations, Department of Toxic Substances Control, January 28.

Cal EPA, 2005. Interim Final Guidance for the Evaluation and Mitigation of Subsurface Vapor Intrusion in Indoor Air. Department of Toxic Substances Control, (revised February 7).

ITRC, 2007. Vapor Intrusion Pathway: A Practical Guide, January.

U.S. EPA, 1988. The Determination of Volatile Organic Compounds in Ambient Air Using Summa™ Passivated Canister Sampling and Gas Chromatographic Analysis, May.

USEPA, 1999. Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition, EPA/625/R-96/010b, January 1999.

U.S. EPA, 2006. Assessment of Vapor Intrusion in Homes Near the Raymark Superfund Site Using Basement and Sub-Slab Air Samples, March 2006. EPA/600/R-05/147.

ATTACHMENT B

SOP FOR THE INDOOR AIR SAMPLING

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LIST OF FORMS
(Following Text)

FORM 1 BUILDING PHYSICAL SURVEY QUESTIONNAIRE

FORM 2 INDOOR AIR SAMPLING FIELD DATA SHEET

FORM 3 INDOOR AIR SAMPLING INSTRUCTIONS TO BUILDING OCCUPANTS

1.0 INTRODUCTION

This Attachment presents the indoor air sampling protocol employed by Conestoga-Rovers & Associates to evaluate the potential presence of volatile organic compounds (VOCs) in indoor air due to subsurface soil and/or groundwater impacts. The protocol presented herein consists of conducting a physical survey of the building to be sampled in conjunction with interviewing building occupants, followed by collection of indoor air samples using 6-liter Summa™ canisters. This indoor air sampling protocol has been developed in consideration of the sampling procedures recommended in the following regulatory guidance documents:

- "*Indoor Air Sampling and Evaluation Guide*" dated April 2002 and prepared by the Massachusetts Department of Environmental Protection (MDEP) (MDEP, 2002)
- "*Guidance for the Evaluation and Mitigation of Subsurface Vapor Intrusion – Interim Final*" dated December 15, 2004 (and revised February 7, 2005) and prepared by the California Environmental Protection Agency (Cal EPA) (Cal EPA, 2004)
- *"Draft Vapor Intrusion Pilot Program Guidance"* dated April 26, 2006 and prepared by the Indiana Department of Environmental Management (IDEM) (IDEM, 2006)
- United States Environmental Protection Agency (USEPA) – Region 5 - *Vapor Intrusion Guidebook,* October 2010 (USEPA, 2010)

Section 2.0 presents the physical building survey to be conducted that will enable a qualitative assessment of factors that potentially could influence indoor air quality. Section 3.0 presents the indoor air sample collection procedure, including quality assurance/quality control (QA/QC) measures and laboratory analytical methodology to be applied in the sample analysis.

2.0 PHYSICAL BUILDING SURVEY

A physical survey will be conducted of the buildings to be sampled. The physical survey will be conducted in conjunction with interviewing the occupants of the buildings. The purpose of the physical survey is to obtain data that will allow a qualitative assessment of factors that potentially could influence indoor air quality. The physical survey includes collecting data on aspects of the building configuration such as building layout, attached garages, utility entrances into the building, ventilation system design, foundation conditions, presence of foundation sump, building material types (e.g., recent carpeting/linoleum and/or painting), location of laundry facilities, etc. The physical survey also includes collecting data related to occupant lifestyle choices that could potentially influence indoor air quality such as use of cleaning products, dry-cleaner use, indoor storage of paints and/or petroleum hydrocarbon products, use of aerosol consumer products, smoking, etc.

The physical survey will be documented by completing the attached Form 1 – Building Physical Survey Questionnaire.

3.0 INDOOR AIR SAMPLE COLLECTION PROCEDURE

Indoor air samples will be collected from the buildings which are or may be occupied that have no slab (e.g., dirt or gravel floor). The indoor air sample will be collected from the lowest floor of the building. An outdoor ambient air sample will be collected concurrently with the indoor air sample from an upwind location on the building property. The indoor and ambient air samples will be collected using a Summa™ canister (6-litre capacity) equipped with a critical orifice flow regulation device sized to allow the collection of an air sample over an 8-hour sampling period. The critical orifice flow regulation device will be supplied and calibrated by the laboratory selected to conduct the sample analysis.

To the extent possible, the indoor air samples will be collected with windows and doors closed to represent appropriately conservative conditions during sampling. If possible, windows and doors should be kept closed for a period of at least 24 hours prior to sample collection. During summer months, air conditioners typically would be operating under closed windows/doors conditions, and the operation of an air conditioner can be allowed during sample collection. This would be representative of season-specific ventilation conditions, and with the expected pattern of operation of the building. Care will be taken to deploy the Summa™ canisters away from the direct influence of any forced air emanating from an air conditioning unit or central air conditioning vents.

The indoor air sampling procedure is described as follows:

- Samples will be collected from an occupied building and as close as practical to the center of the area, but away from high traffic areas to minimize the potential for disturbances during sample collection. Typically, sample canisters will be located between 1 to 1.5 meters above floor level.
- For each ambient air sample, a suitable upwind location (selected to minimize the potential for disturbances during sample collection) will be selected. The ambient air sample will be collected a minimum of 1 meter above grade (if possible) and located to minimize the potential for disturbance of the canister while providing protection from weather effects.
- Air sample canisters will be labeled with a unique sample designation number. Both the sample number and the sample location information will be recorded on the attached Form 2 – Indoor Air Sampling Field Data Sheet.
- The Summa™ canister vacuum will be measured immediately prior to canister deployment and recorded on Form 2 – Indoor Air Sampling Field Data Sheet.

- The critical orifice flow controller will be installed, as supplied by the laboratory, on the canister and the canister will be opened fully at the beginning of sample collection period and start time recorded on Form 2 – Indoor Air Sampling Field Data Sheet.
- At the start and the end of the 8-hour sample period, a portable photoionization detector (PID) will be used to screen for VOC presence in the sample area. Results of the PID monitoring were recorded on Form 2 – Indoor Air Sampling Field Data Sheet.
- Other data recorded on Form 2 – Indoor Air Sampling Field Data Sheet will include: outside and interior temperatures both at the start and end of the sample period, equipment serial numbers, sampler name, and any comments.
- Following equipment setup, the building occupant will be given the list of instructions to follow while the Summa™ canister sample is being taken in the building. The instructions are listed in the attached Form 3 - Indoor Air Sampling Instructions to Building Occupants. The date and completion time of the 8-hour sample period will be written on Form 3 and the occupant will be instructed that the sampling team would be back to pick up the canister after approximately 8 hours.
- The canister valve will be closed fully at the end of the sample period (after 8 hours) and the end time recorded on the field data sheet. If there is evidence of canister disturbance during the sample collection, this will be recorded on Form 2 – Indoor Air Sampling Field Data Sheet.
- The Summa™ canister vacuum will be measured immediately after canister retrieval at the end of the 8-hour sample period and recorded on the field data sheet. Any samples where the canister reached atmospheric pressure will be rejected and the canisters returned for cleaning. The minimum vacuum required to be considered a valid sample will be 1 to 2 inch Hg vacuum. Once the vacuum is measured, the safety cap will be securely tightened on the inlet of the Summa™ canister prior to shipment to the laboratory under CRA chain of custody procedures. The requirement for residual vacuum retained in the canister following sample collection is to ensure that a driving force was maintained to collect a steady flow rate until the end of the sampling event.
- The Summa™ canister vacuum will be measured by the laboratory immediately prior to sample analysis and recorded on the analytical data report.
- All canisters will be cleaned in accordance with United States Environmental Protection Agency (USEPA) Method TO-15 and documentation of the cleaning activities will be obtained from the laboratory.

3.1 QUALITY ASSURANCE/QUALITY CONTROL

Quality Assurance/Quality Control (QA/QC) samples will collected during the indoor air sampling. QA/QC samples will include:

- the ambient air sample
- one duplicate

3.2 ANALYTICAL METHOD/LABORATORY

The soil vapor samples will be analyzed by a certified laboratory using the USEPA TO-15 gas chromatograph/mass spectrometer (GC/MS) methodology.

3.3 DATA VALIDATION

A data validation for the air sample result will be conducted by CRA.

3.4 CANISTER CLEANING

Canister cleaning was completed in accordance with the applicable sections of Method TO-15.

4.0 REFERENCES

Cal EPA, 2004. Guidance on the Evaluation and Migration of Subsurface Vapor Intrusion to Indoor Air – Interim Final, Department of Toxic Substances Control, California Environmental Protection Agency, December 15 (revised February 7, 2005).

IDEM, 2006. Draft Vapor Intrusion Pilot Program Guidance. Indiana Department of Environmental Management, April 26.

MDEP, 2003. Indoor Air Sampling and Evaluation Guide, WSC Policy #02-430, Office of Research and Standards, Massachusetts Department of Environmental Protection, April.

USEPA, 2010. Region 5 - Vapor Intrusion Guidebook, United States Environmental Protection Agency .

FORM 1: BUILDING PHYSICAL SURVEY QUESTIONNAIRE

Address: _____

Building Owner: _____

Occupant Name: _____

Date: _____ Time: _____ Inspector: _____ Sample No.: _____

Contact Name: _____ Phone Number: _____

How long have you lived/worked in this home/building? _____

Occupation: _____

Number of Occupants Adults: _____
Children: _____

BUILDING TYPE: One story ____ Two storey _____ Brick _____ Siding _____ Stucco ___

DESCRIBE BUILDING: _____ **YEAR CONSTRUCTED**: _____

WEATHER SEALS: General Condition: Good _____ Fair _____ Poor _____

BASEMENT:	None	❑	Finished	Unfinished	Depth below reference point (meters)
	Partial	❑	❑	❑	_____
	Full	❑	❑	❑	_____
	Crawl space	❑	na	na	_____

Number of floors at or above grade: _____

Depth of basement below grade: _____ ft. Basement Size: _____ ft^2

Foundation construction: Poured concrete ❑ Cinder block ❑ Stone ❑

Any visual evidence of leakage through basement walls or floor ❑

Floor Construction: Poured concrete ❑ Wood ❑ Earth ❑ Brick ❑ Other: _____

Floor condition (cracks, drains): _____

Condition at floor/wall joint (if visible): _____

Any exterior openings from the basement:

❑ Vents

❑ Fans

❑ Windows

- ❑ Wall openings
- ❑ Utility pipe penetrations
- ❑ Other: _____

Type of ground cover outside of building: grass / concrete / asphalt / other (specify): _____

Sub-slab vapor/moisture barrier in place? Yes / No / Don't know

Type of barrier: _____

Do you have a sump?: Yes ❑ No ❑

Where: _____

If yes, sealed ❑ open ❑ NA ❑

If yes, is there water in the sump?: Yes ❑ No ❑

Is this building serviced with municipal water? Yes ❑ No ❑

Do you have a water well?: Yes ❑ No ❑ Don't know ❑

Well location: _____

Do you drink the water obtained from the well? _____

What do you use the well for?: _____

Do you have a cistern?: Yes ❑ No ❑

If yes, describe its location: _____

Do you have a septic system?: Yes ❑ No ❑

If yes, describe its location: _____

If yes, describe how septic system is cleaned: _____

_____

Have there ever been a fire in the building?: Yes ❑No ❑

If yes, describe its location and extent: _____

Is there a laundry room located inside the house/building?: Yes ❑ No ❑

If yes, describe its location: _____

FURNACE: Location:_____

Type:	gas ❑	Forced air ❑
	oil ❑	hot water ❑
	electric ❑	other _____

Does furnace have outside combustion air vent? _____

Do you have a fireplace? Yes ❑ No ❑

Does the fireplace have an outside combustion air vent? Yes ❑ No ❑

Do you use kerosene space heaters? Yes ❑ No ❑

FORM 1: BUILDING PHYSICAL SURVEY QUESTIONNAIRE

AIR CONDITIONER: None _____ Central _____Room _____
(If yes, which rooms and capacities?) _____

RADON SYSTEM: ❑ Yes ❑ No

GARAGE: Do you have an attached garage? ❑ Yes ❑ No

1. When was the last time dry-cleaned clothes were brought into the house/building?

   ❑ 0 to 5 days ago ❑ 6 to 10 days ago ❑ More than 10 days ago ❑ Don't dry-clean

2. When was your carpet installed?

   ❑ In the last six months ❑ More than six months ago ❑ No Carpet

3. When was the last time your carpet was cleaned?

   ❑ In the last six months ❑ More than six months ago ❑ Never

4. Do you have any spot removers in the house?

   ❑ Yes ❑ No Details: _____

5. Do your hobbies include model building, arts and crafts, model railroading, or others that require paints, thinners, or glue?

   ❑ Yes ❑ No Details: _____

6. Do you perform automotive or other vehicle maintenance or repair at home?

   ❑ Yes ❑ No Details: _____

7. Please review the following list and check items you know are in your home

   ❑ Latex caulk
   ❑ Latex paint
   ❑ Vinyl cove molding
   ❑ Linoleum tile
   ❑ Black rubber molding
   ❑ Vinyl edge molding
   ❑ Polystyrene foam insulation
   ❑ Adhesive removers

- ❑ Aerosol spray paints
- ❑ Other paints
- ❑ Air fresheners
- ❑ Degreasers
- ❑ Deodorants
- ❑ Disinfectants
- ❑ Furniture Polish
- ❑ Solvents
- ❑ Caulking

8. Do you have pesticides in your home/building?

❑ Yes ❑ No ❑ Unsure

9. Do you have any spray insecticides in your home/building?

❑ Yes ❑ No ❑ Unsure

10a. Have you painted any area of the interior of your home/building in the last 12 months?

❑ Yes ❑ No

10b. If yes, please indicate what paint you used

- ❑ Enamel
- ❑ Vinyl
- ❑ Latex
- ❑ Other

11a. Have you painted the exterior of your building in the last 12 months? ❑ Yes ❑ No

11b. If yes, please indicate what paint you used

- ❑ Enamel
- ❑ Vinyl
- ❑ Latex
- ❑ Other

12. Where do you store your paint, thinner, pesticides, insecticides?

	Paint	*Thinner*	*Pesticides*	*Insecticides*
Garage	❑	❑	❑	❑
Basement	❑	❑	❑	❑
Storage shed	❑	❑	❑	❑
Other	❑	❑	❑	❑

❑ I don't store these items at home

13. Have you purchased one of the following items in the last 12 months?

❑ Rubberized door mat ❑ Computer ❑ Wiring
❑ Plastic shower curtain ❑ Printer ❑ Linoleum
❑ Wood stains or paint ❑ VCR

14. Do you have a computer printer in your home/building?

❑ Yes ❑ No

15. Do you have a VCR in your home/building?

❑ Yes ❑ No

16. Do you use cleaners to maintain your VCR/building?

❑ Yes ❑ No

If yes, what type? _____

17. Do you have pets residing in this building?

❑ Yes ❑ No

If yes, what type? _____

If yes, number _____

18. Does anyone in the building smoke? ❑ Yes ❑ No

19. Questions asked by Occupant that require follow-up.

_____

_____

_____

_____

FORM 2: INDOOR AIR SAMPLING FIELD DATA SHEET

A) General Information

Sample Identification Number: _____
Site Address: _____
Sample Canister Location: _____

Sample source: Indoor Air / Sub-Slab / Near Slab Soil Gas / Exterior Soil Gas
Sample Date: _____ Sampler: _____
Sample Time: Start: _____ Stop: _____
Shipping Date: _____

Canister Type: 400 mL – 1.0 L Summa Canister/6 L Summa Canister/Other (specify):
_____
Canister Serial No.: _____
Flow Controller Serial No.: _____

Were "Instructions for Occupants" followed?
❑ Yes ❑ No

B) Sampling Information

	Start		Stop	
	Ambient	Interior	Ambient	Interior
Temperature	_____	_____	_____	_____

	Start	Stop
Canister Pressure Gauge Reading:	_____	_____
Time:	_____	_____
PID Reading (ppm):	_____	_____
Basement Depth (ft below grade):	_____	
Window Marked:	Yes/No	

Was there significant precipitation within 12 hours prior to (or during) the sampling event?
❑ Yes ❑ No

Describe the general weather conditions:_____
_____
_____

Provide Drawing of Sample Location(s) in Building

C) Comments

1. The duration of this test is approximately 8 hours.

2. The canister is made of clean stainless steel. It does not contain any moving parts or chemicals.

3. Do not handle or move a canister during testing.

4. Do not smoke around the canister.

5. To the extent possible, leave doors and windows closed during testing.

6. To the extent possible, do not use paint, solvents, glues, and spray cans during testing.

7. If possible, do not bring dry cleaning into the building during the testing.

8. We will be back tomorrow to pick up the canister about this time.

Canister pick up: Day_____

Time_____

Thank you for your cooperation.

ATTACHMENT C

SUB-SLAB SOIL VAPOR AND
INDOOR AIR SAMPLING ACTIVITIES

SUB-SLAB SOIL VAPOR AND INDOOR AIR SAMPLING ACTIVITIES

The objectives of the sub-slab soil gas sampling are as follows:

- Determine whether contaminant concentrations pose more than a 1×10^{-4} cancer risk or a hazard index (HI) greater than 1.0 through the vapor intrusion (VI) pathway to current or potential future receptors
- Determine whether concentrations of combustible gases within a structure exceed 10 percent of the lower Explosive Limit (LEL) for methane)
- Identify buildings where indoor air sampling is required based on the sub-slab sample results

Sub-slab soil vapor probes will be installed in accordance with the Vapor Intrusion Investigation Work Plan, dated December 17, 2010. Sub-slab soil vapor probes will be installed beneath the following existing on-Site structures:

A. *Structures On Site West of Dryden Road:*
3 building structures on Lot 5054
3 building structures on Lot 5171
2 building structures on Lot 5172
1 building structure on Lot 5174
1 building structure on Lot 5175, and

B. *Structures On Site or Adjacent to Site Along East River Road:*
4 building structures on Lot 4610 (Barnett; on-Site)
2 building structures on Lot 3207
1 residence on Lot 3253; and
1 building structure on Lot 3254.

Prior to conducting the sub-slab soil vapor sampling, CRA will visually inspect of the Lots in question and document the number and type of buildings present on each Lot in order to ensure that all buildings that are or may be occupied are included in the sampling program.

Prior to installing the sub-slab soil vapor probes, a survey will be conducted of each building, to identify potential preferential pathways for vapor migration under the building. Details of the building survey are included in the Vapor Intrusion Investigation Work Plan. If any structure on or adjacent to the Site that is or may be occupied has no slab (e.g., dirt or gravel floor), indoor air samples will be collected. For any location where an indoor air sample is collected, CRA will also install a soil vapor probe screened between 3 and 5 feet below ground

surface in accordance with CRA's SOP [Appendix J-F-11 of the Field Sampling Plan (FSP)] in order to attempt to correlate indoor air concentrations to concentrations of contaminants in soil vapor near the structure. The soil vapor probes will be installed immediately adjacent to the side of the building closest to the most likely source of any soil vapor impacts. In addition, where indoor air samples are collected, CRA will also collect ambient air samples immediately adjacent to the structure as per CRA's SOP.

CRA's standard operating procedure (SOP) for installing sub-slab probes and collecting sub-slab vapor samples are in Attachment A to the Vapor Intrusion Investigation Letter Work Plan (addendum to the FSP). CRA's SOP for indoor air sampling is in Attachment B to the Vapor Intrusion Letter Work Plan (addendum to the FSP).

If collected, sub-slab soil gas samples will be analyzed for benzene, toluene, ethylbenzene, and xylenes (BTEX), along with chlorinated VOCs including perchloroethylene (PCE), trichloroethylene (TCE), cis/trans-1,2-dichloroethylene (1,2-DCE), 1,1-dichloroethylene (1,1-DCE), and VC in accordance with the USEPA Toxic Organics-15 (TO-15) parameter list.