SAP Worksheet #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
September 30, 2010

UFP-SAP for Operations, Maintenance, and Monitoring of the
Groundwater Treatment Plant
GM-38 Area, Naval Weapons Industrial Reserve Plant
Bethpage, New York

Prepared for:
Department of the Navy
Naval Facilities Engineering Command
Mid-Atlantic
NAVFAC, MIDLANT OPNEEV
Environmental Restoration
Building Z-144, Room 109
9742 Maryland Avenue
Norfolk, VA 23511-3095

Prepared by:
Tetra Tech EC, Inc.
820 Town Center Drive, Suite 100
Langhorne, PA 19047

Prepared under:
Contract No. N62472-99-D-0032
Contract Task Order Number 0096

Review Signature:  _____
Tetra Tech EC, Inc. QAM/Program QAM
Jonathan Dziekan

Review Signature: _____
NAVFAC QAO/Chemist
Jonathan Tucker

Approval Signature:  _____
Tetra Tech EC, Inc. Project Manager
Stavros Patselas

Approval Signature: _____
NAVFAC Remedial Project Manager
Lora Fly

Approval Signature: _____
NYSDEC Project Engineer
Steven Scharf

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EXECUTIVE SUMMARY

This Uniform Federal Policy (UFP)-Sampling and Analysis Plan (SAP) describes the operation, maintenance, and monitoring (OM&M) activities associated with the groundwater treatment plant (GWTP) at the Naval Weapons Industrial Reserve Plant (NWIRP), GM-38 Area, in Bethpage, New York. The UFP-SAP was prepared by Tetra Tech EC, Inc. (TtEC) on behalf of Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic under Contract Number N62472-99-D-0032, Contract Task Order Number 0096.

As stated in the Navy's Record of Decision (ROD), the purpose of the groundwater treatment system is to "Eliminate, to the extent practical, site-related contaminants from the affected public water supplies and to prevent, to the extent practical, the future contamination of public water supplies through the implementation of the offsite groundwater remediation." The treatment system has been designed for a 5 to 10-year operational life. It is not intended to remediate groundwater contamination in the local aquifer to non-detectable levels. Rather, the intent of the system is to remove mass, reduce elevated volatile organic compound (VOC) levels to levels similar to those in the surrounding aquifer, and minimize the impacts on water supply wells and currently unaffected portions of the aquifer.

Several sampling and monitoring programs will be conducted as part of the GWTP operations. These include:

- sampling and monitoring of influent, effluent, and intermediate process streams during the start-up period;
- sampling and monitoring of influent, effluent, and intermediate process streams during the prove-out period;
- sampling and monitoring of process streams (including influent, effluent, and intermediate) for routine operations; and
- sampling and monitoring of groundwater.

During the start-up period, various process streams will be sampled and/or monitored. The purpose of this sampling and monitoring is for tracking and documenting GWTP operation performance as well as for regulatory compliance. The start-up period is defined as the first 30 days of operations. During the system start-up period, all samples will be collected on a weekly basis, including those required for regulatory purposes as well as those not required for regulatory compliance purposes but are beneficial to ensure the GWTP is operating properly. Aqueous and air samples will be collected from the process streams via sample ports.

Following the start-up period, a prove-out period will begin and extend until the GWTP has been functional for an additional 5 months. The same process streams will be sampled as during the start-up period, and the purpose of this sampling and monitoring is to ensure the GWTP is operating in accordance with the design specifications and that effluent streams meet all regulatory and disposal facility requirements. During the system prove-out period, all samples will be collected on a weekly basis, including those required for regulatory purposes as well as those not required for regulatory compliance purposes but are beneficial to ensure the GWTP is operating according to design specifications. Based on the data that is collected during the start-up and prove-out periods, after consultation with the Navy Remedial Project Manager (RPM), TtEC may decide to reduce the frequency of sampling and analyses for the influent process water and some of the intermediate process streams. Aqueous and air samples will be collected from the process streams via sample ports.

During routine operations, the same process streams as those sampled during the start-up and prove-out periods will be sampled and/or monitored. It should be noted that most of this sampling and monitoring is for the purpose of tracking and documenting the performance of plant operations and not for regulatory compliance reporting purposes. Only the process effluent streams will be sampled for regulatory compliance purposes for the parameters identified by New York State Department of Environmental Conservation (NYSDEC) and the disposal facilities at the designated frequency. Based on experience

gained in operating the GWTP, following the start-up and prove-out periods as discussed below, and after consultation with the Navy RPM, TtEC may reduce the frequency of sampling and analyses for the influent process water and some of the intermediate process streams for plant operations. All analytical parameters for regulatory compliance will be collected monthly (samples for pH will be collected weekly) and analyzed at a laboratory certified by the State of New York for these parameters. Samples from recovery wells RW-1 and RW-3 as well as the treated effluent will be collected once every two weeks.

Sampling and monitoring of the groundwater from the 12 existing and proposed monitoring wells will be performed throughout the period of operation of the GWTP and for two years beyond the shut-down of GWTP operations to determine the effectiveness of the remediation activities and monitor the hydraulic containment and capture of the groundwater “hot spot” by the recovery wells. Water level measurements will be performed in all 12 monitoring wells on a quarterly basis. In addition, samples for water quality monitoring will be collected from eight of the 12 wells on a quarterly basis for the first two years from the start of GWTP operations, on a semi-annual basis for years three and four, and then on an annual basis from the fifth year onwards. It was deemed unnecessary to collect samples from all 12 wells because some of them are in close proximity and are therefore expected to have the same water quality.

The final determination to take the GWTP off-line will be made by the Navy in consultation with NYSDEC. When concentrations of chlorinated VOCs in the GM-38 Area groundwater “hot spot” are equal to those concentrations in the surrounding aquifer, TtEC will make the recommendation to the Navy that operations at the GWTP be terminated. With consent from the Navy and NYSDEC, sampling and monitoring of the groundwater quality will continue for two years (on a quarterly basis) beyond the shut-down of the GWTP operations.

SAP Worksheets

List of Figures

Appendices

ACRONYMS

AA	Atomic Absorption
AES	Atomic Emission Spectrometry
bgs	Below Ground Surface
B.S.	Bachelor of Science
COC	Chain of Custody
CFR	Code of Federal Regulations
CIH	Certified Industrial Hygienist
DI	Deionized
DoD	Department of Defense
DOT	Department of Transportation
DQI	Data Quality Indicator
DQO	Data Quality Objective
EFANE	Engineering Facility Northeast
ELAP	Environmental Laboratory Accreditation Program
EPA	Environmental Protection Agency
FOL	Field Operations Leader
FT	Feet
FTMR	Field Task Modification Request
GC/MS	Gas Chromatograph/Mass Spectrometer
GWTP	Groundwater Treatment Plant
HASP	Health and Safety Plan
HDPE	High Density Polyethylene
HSM	Health and Safety Manager
hr	Hour
ICP	Inductively Coupled Plasma
L	Liter
lb	Pound
LCS	Laboratory Control Sample
LDR	Linear Dynamic Range
LGAC	Liquid-phase Granular Activated Carbon
LIMS	Laboratory Information Management Systems
LQAP	Laboratory Quality Assurance Plan
m^3	Cubic Meter
MDL	Method Detection Limit
mg	Milligram
MPC	Measurement Performance Criteria
MS	Matrix Spike
M.S.	Master of Science
MSD	Matrix Spike Duplicate
NA	Not Applicable
NAVFAC	Naval Facilities Engineering Command
NFESC	Naval Facilities Engineering Service Center
NGC	Northrop Grumman Corporation
NIST	National Institute of Standards and Technology
NTU	Nephelometric Turbidity Unit
NWIRP	Naval Weapons Industrial Reserve Plant
NYSDEC	New York State Department of Environmental Conservation
OM&M	Operations, Maintenance, and Monitoring
OSHA	Occupational Safety and Health Administration
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PAL	Project Action Limit
PCB	Polychlorinated Biphenyl
PCE	Tetrachloroethene

PESM	Program Environmental and Safety Manager
Ph.D.	Doctor of Philosophy
PM	Project Manager
PQO	Project Quality Objective
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCS	Quality Control Sample
QL	Quantitation Limit
QSM	Quality Systems Manual
%R	Percent Recovery
RAC	Remedial Action Contract
RCRA	Resource Conservation and Recovery Act
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPDES	State Pollution Discharge Elimination System
SSHO	Site Safety and Health Officer
SVOC	Semi-Volatile Organic Compound
TAT	Turnaround Time
TBD	To Be Determined
TCE	Trichloroethene
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TSS	Total Suspended Solids
TtEC	Tetra Tech EC, Inc.
UFP	Uniform Federal Policy
ug (µg)	Microgram
VGAC	Vapor-phase Granular Activated Carbon
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

SAP Worksheet #2 -- SAP Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: **GM-38 Area, Naval Weapons Industrial Reserve Plan (NWIRP)**
Operable Unit: **OU2**
Contractor Name: **Tetra Tech EC, Inc. (TtEC)**
Contract Number: **N62472-99-D-0032**
Contract Title: **U.S. Navy Southwest Remedial Action Contract (RAC)**
Work Assignment Number (optional): CTO 0096

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) (EPA, 2005) and United States Environmental Protection Agency (EPA) Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (2002). Identify any additional guidance used to prepare SAP: None

2. Identify regulatory program: New York State Pollution Discharge Elimination System (SPDES)

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Email correspondence and telephone conversations with the Navy to discuss the Operations, Maintenance, and Monitoring (OM&M) Plan	May 2009

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current remediation.

Title	Date
No other sampling and analysis plans have been developed for the OM&M phase of the remediation.	Not Applicable (NA)

6. List organizational partners (stakeholders) and connection with lead organization:
New York State Department of Environmental Conservation (NYSDEC) (regulatory oversight) and Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic (property owner).

7. Lead organization (see Worksheet #7 for detailed list of data users) NAVFAC Mid-Atlantic

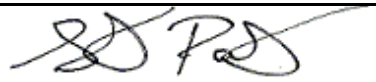
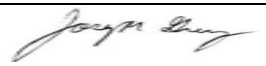
8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:
Cross-walk omitted, not needed.

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

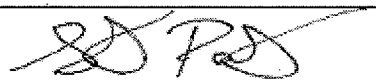
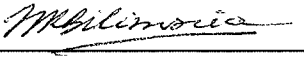
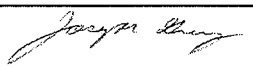
Name of SAP Recipient	Title/Role	Organization	Telephone Number	Email Address or Mailing Address	Document Control Number
Lora Fly	Remedial Project Manager (RPM)	NAVFAC Mid-Atlantic	757-444-0781	lora.fly@navy.mil	Not Applicable (NA)
Steven Scharf	Project Engineer	NYSDEC	518-402-9620	sxscharf@gw.dec.state.ny.us	NA
Stavros Patselas	Project Manager (PM)	TtEC	215-702-4099	stavros.patselas@tetratech.com	NA
Maheyar Bilimoria	OM&M Engineer	TtEC	215-702-4049	maheyar.bilimoria@tetratech.com	NA
TBD	Field Operations Leader (FOL)	TtEC	TBD	TBD	NA
Joseph Grey	Site Safety Officer(SSHO)	TtEC	215-702-4000	joseph.grey@tetratech.com	NA
Lynn Arabia	Project Chemist	TtEC	973-630-8356	lynn.arabia@tetratech.com	NA
Veronica Bortot	Project Manager	Test America – Pittsburgh	412-963-2435	Veronica.Bortot@Test Americainc.com	NA
Ausha Scott	Project Manager	Air Toxics, Ltd.	916-985-1000	ascott@airtoxics.com	NA

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Name	Organization/Title/Role	Telephone Number (optional)	Signature/Email Receipt	SAP Section Reviewed	Date SAP Read
Stavros Patselas	TtEC/Project Manager	215-702-4099		All	8/28/09
Maheyar Bilimoria	TtEC/OM&M Engineer	215-702-4049		All	8/21/09
Jonathan Dziekan	TtEC/ Program Quality Assurance Manager (QAM)	215-702-4023		All	8/28/09
Lynn Arabia	TtEC/ Project Chemist	973-630-8356		All	8/19/09; 9/15/10
Fred Mattison	ECOR Solutions, Inc. / Plant Operator	610-840-9200			
TBD	TtEC / FOL	TBD			
Joseph Gray	TtEC / SSHO	215-702-4000		All	8/27/09
Veronica Bortot	Test America – Pittsburgh / Project Manager	412-963-2435			
Ausha Scott	Air Toxics, Ltd. / Project Manager	916-985-1000			

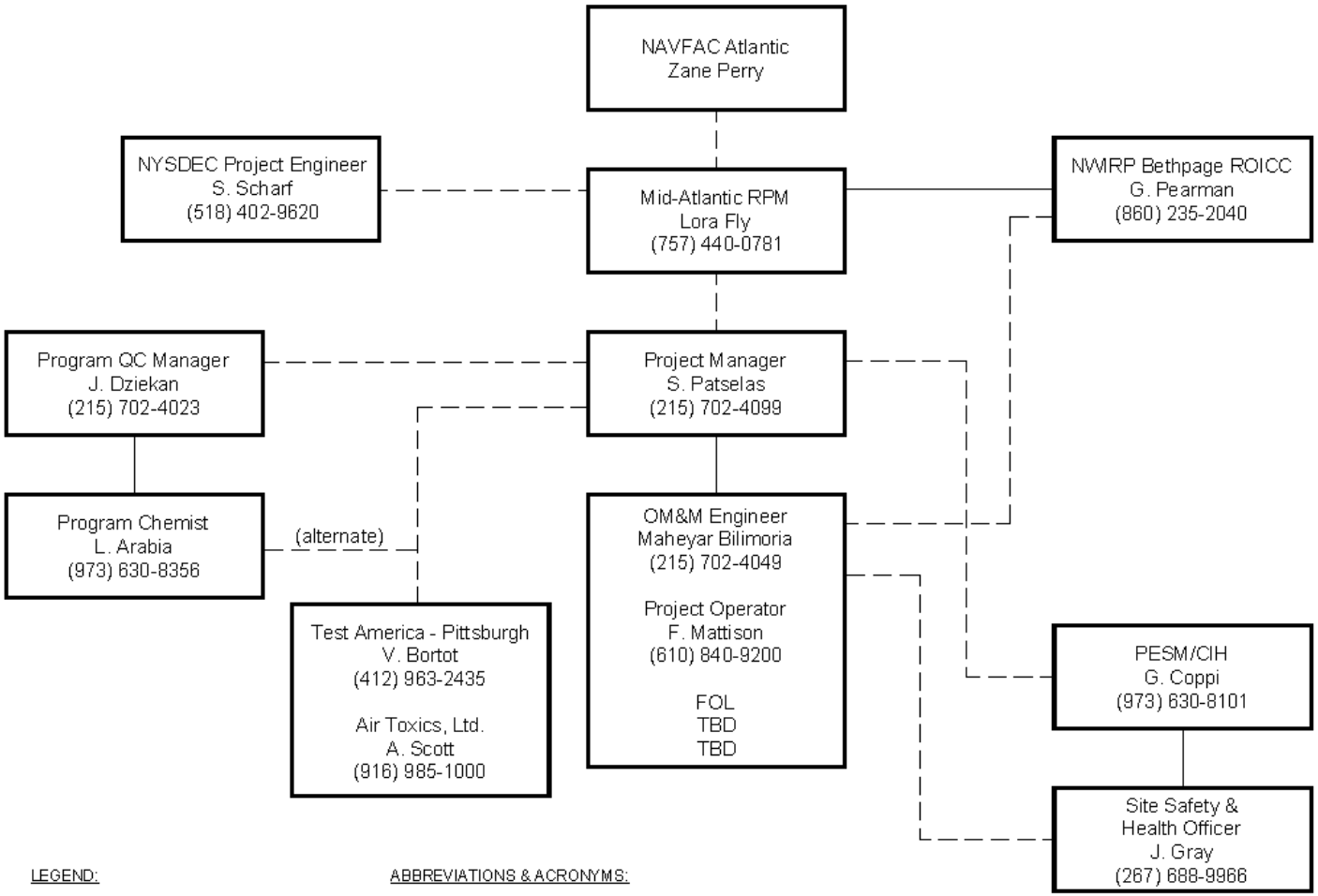
SAP Worksheet #4 -- Project Personnel Sign-Off Sheet

(UFP-QAPP Manual Section 2.3.2)

Name	Organization/Title/Role	Telephone Number (optional)	Signature/Email Receipt	SAP Section Reviewed	Date SAP Read
Stavros Patselas	TtEC/Project Manager	215-702-4099		All	8/28/09
Maheyar Bilimoria	TtEC/OM&M Engineer	215-702-4049		All	8/21/09
Jonathan Dziekan	TtEC/ Program Quality Assurance Manager (QAM)	215-702-4023		All	8/28/09
Lynn Arabia	TtEC/ Project Chemist	973-630-8356		All	8/19/09; 9/15/10
Fred Mattison	ECOR Solutions, Inc. / Plant Operator	610-840-9200			
TBD	TtEC / FOL	TBD			
Joseph Gray	TtEC / SSHO	215-702-4000		All	8/27/09
Veronica Bortot	Test America – Pittsburgh / Project Manager	412-963-2435			
Ausha Scott	Air Toxics, Ltd. / Project Manager	916-985-1000			

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)



LEGEND:

- — — — IN REGULAR CONTACT AND COORDINATION
- ——— DIRECTLY REPORTS TO ABOVE

ABBREVIATIONS & ACRONYMS:

CIH	Certified Industrial Hygienist
FOL	Field Operations Lead
NAVFAC	Naval Facilities Engineering Command
NWIRP	Naval Weapons Industrial Reserve Plant
NYSDEC	New York State Department of Environmental Conservation
OM&M	Operations, Maintenance, and Monitoring
PESM	Program Environmental and Safety Manager
QC	Quality Control
ROICC	Resident Officer in Charge of Construction
RPM	Remedial Project Manager

SAP Worksheet #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Driver	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
SAP Amendments	RPM	Lora Fly	757-444-0781	Immediately informs TtEC PM. Document via Field Task Modification Request (FTMR) form; complete within 1 week or less.
Changes in Schedule	TtEC PM	Stavros Patselas	215-702-4099	Informs Navy RPM via schedule impact letter as soon as impact is realized. Schedule revised within 1 week or less.
Unanticipated field conditions that would require a change in plan and result in FTMRs	ECOR Plant Operator TtEC FOL TtEC Project Manager	Fred Mattison TBD Stavros Patselas	610-840-9200 TBD 215-702-4099	Plant Operator/FOL immediately informs PM; PM informs RPM; RPM issues scope change if warranted; scope change to be implemented before work is executed. Document via FTMR form (within 1 week or less). Complete within time frame as per agreement with Navy after authorization.
Conditions adverse to quality or health and safety	ECOR Plant Operator TtEC FOL TtEC PM TtEC QAM TtEC Program Environmental and Safety Manager (PESM) TtEC Site Safety and Health Officer (SSHO) Navy RPM	Fred Mattison TBD Stavros Patselas Jonathan Dziekan Grey Coppi Joe Gray Lora Fly	610-840-9200 TBD 215-702-4099 215-702-4023 973-630-8101 267-688-9966 757-444-0781	Responsible party immediately informs subcontractors, the Navy, and Project Team to stop work. Re-initiate work upon corrective action within 1 week or less (as possible).
Analytical data quality issues	Test America – Pittsburgh PM Air Toxics, Ltd. PM TtEC Project Chemist Navy Chemist	Veronica Bortot Ausha Scott Lynn Arabia Jon Tucker	412-963-2435 916-985-1000 973-630-8356 TBD	Immediately notify TtEC Project Chemist. Notify data review staff and TtEC PM if necessary. Notify Navy RPM and Chemist, if necessary. Resolve within 1 week or less.

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table
(UFP-QAPP Manual Section 2.4.3)

Data users: TtEC, NYSDEC (regulatory oversight), and Navy (property owner).

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Stavros Patselas	PM	TtEC	Oversees project, financial, schedule, and technical day-to-day management of the project. • Ensures timely resolution of project-related technical, quality, and safety questions associated with TtEC operations. • Functions as the primary TtEC interface with the Navy RPM, regulators, TtEC field and office personnel, and laboratory points of contact. • Ensures that TtEC health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories. • Monitors and evaluates all TtEC subcontractor performance. • Coordinates and oversees work performed by TtEC field and office technical staff (including data review, data interpretation, and report preparation). • Coordinates and oversees maintenance of all TtEC project records. • Coordinates and oversees review of TtEC project deliverables. • Prepares and issues final TtEC deliverables to the Navy.	B.S., Civil and Environmental Engineering, 13 years environmental experience
Maheyar Bilimoria	OM&M Engineer	TtEC	Oversees operation, maintenance and operation of the GWTP in accordance with design. • Ensures timely resolution of project-related technical questions associated with GWTP operations. • Functions as the primary TtEC interface with the TtEC field and office personnel and the plant operator. • Oversee plant operation subcontractor. • Coordinates and oversees work performed by TtEC field	Ph.D., Chemical Engineering, 35 years environmental experience

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
			and office technical staff (including data review, data interpretation, and report preparation). • Reviews TtEC project deliverables.	
Joseph Gray	SSHO	TtEC	Supervises, coordinates, and performs field sampling activities • Ensures that all health and safety requirements are implemented. • Functions as the on-site communications link between field staff members and TtEC PM. • Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples. • Oversees the mobilization and demobilization of all field equipment and subcontractors. • Coordinates and manages the field technical staff. • Adheres to the work schedules provided by the TtEC PM. • Ensures the proper maintenance of site logbooks, field logbooks, and field recordkeeping. • Initiates FTMRs (field change orders) when necessary. • Identifies and resolves problems in the field via consultation with the PM, implements and documents corrective action procedures, and provides communication between the field team and project management.	BS, Environmental Engineering, 16 years environmental experience
Fred Mattison	Plant Operator	ECOR Solutions, Inc.	Remotely operates the plant, performs minor maintenance, oversees subcontractors during major maintenance or upgrade activities, and responds to alarm conditions or plant shut-downs. • Ensures that all health and safety requirements are implemented. • Adheres to the work schedules provided by the TtEC PM. • Ensures the proper maintenance of the GWTP recordkeeping.	TBD

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Lynn Arabia	Project Chemist	TtEC	Coordinates analyses with laboratory chemists, ensures the scope is followed, provides quality assurance reviews of data packages, and communicates with TtEC staff. • Ensures that the project meets objectives from the standpoint of laboratory performance. • Provides technical advice to the TtEC team on matters of project chemistry. • Monitors and evaluates subcontractor laboratory performance. • Ensures timely resolution of laboratory-related technical, quality, or other issues effecting project goals. • Functions as the alternate interface with the subcontracted laboratory and the TtEC PM. • Coordinates and oversees work performed by the subcontracted laboratory. • Coordinates and oversees review of laboratory deliverables. • Recommends appropriate laboratory corrective actions. • Reviews chemistry and validation information in SAP.	B.S., Chemistry, 17 years environmental experience
Grey Coppi, CIH	HSM	TtEC	Oversees Remedial Action Contract (RAC) Health and Safety Program.	M.S, Environmental Health Science B.S., Health Science, 23 years of environmental experience
Jonathan Dziekan	QAM	TtEC	Reviews SAP, oversees preparation of laboratory scope, coordinates with lab, and oversees data quality review. Ensures quality aspects of the RAC program. • Develops, maintains, and monitors Quality Assurance (QA) policies and procedures. • Provides training to TtEC staff in QA/Quality Control (QC) policies and procedures. • Conducts systems and performance audits to monitor compliance with environmental regulations, contractual requirements, QAPP requirements, and corporate policies and procedures.	M.S., Civil and Environmental Engineering; 9 years of environmental experience

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
			• Audits project records. • Monitors subcontractor quality controls and records. • Assists in the development of corrective action plans and ensuring correction of non-conformances reported in internal or external audits. • Ensures that this SAP meets TtEC, Navy, and NYSDEC requirements. • Prepares QA reports for management.	
Veronica Bortot	PM	Test America – Pittsburgh	Coordinates analyses with laboratory chemist, ensures that scope is followed, performs quality assurance reviews of data packages, ensures current certification with Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP), and communicates with TtEC.	TBD
Ausha Scott	PM	Air Toxics, Ltd.	Coordinates analyses with laboratory chemist, ensures that scope is followed, performs quality assurance reviews of data packages, ensures current certification with DoD ELAP, and communicates with TtEC.	TBD

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(b)(4). Safety requirements will be addressed in greater detail in the TtEC Health and Safety Plan (HASP). The HASP will be submitted as a separate document.

The selected analytical laboratories (Test America – Pittsburgh and Air Toxics, Ltd.) will successfully complete the laboratory evaluation process required as part of the Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) and described in the DoD Quality Systems Manual (QSM) dated April 2009. Laboratory ELAP certifications are provided in Appendix C for Test America – Pittsburgh and Appendix D for Air Toxics, Ltd.

In addition, the Operator of the groundwater treatment plant (GWTP) will be required to participate in a field training program given by TtEC and/or selected equipment manufacturer representatives. The training will address equipment operation, maintenance, safety requirements and troubleshooting, and other subjects required to properly operate the GWTP.

The training requirements of 6 NYCRR Part 650 will be applicable for the plant operator.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Naval Weapons Industrial Reserve Plant
Site Name: GM-38 Area
Site Location: Bethpage, New York
Projected Date(s) of Remediation: 2009 through 2014
Project Manager: Stavros Patselas

Date(s) of Session: Emails/Telephone Conversations; May 2009
Scoping Session Purpose: Identify OM&M Plan Elements and Information Required to Complete OM&M Plan.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
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Consensus Decisions: Prepared suggested Table of Contents for OM&M Plan. Identified specific information / documents required to prepare and complete draft OM&M Plan.

SAP Worksheet #10 -- Problem Definition
(UFP-QAPP Manual Section 2.5.2)

10.1 SITE DESCRIPTION

10.1.1 Site Location

NWIRP Bethpage is located in east-central Nassau County, Long Island, New York, approximately 30 miles east of New York City. The Navy's property totaled approximately 109.5 acres and was formerly a Government Owned Contractor-Operated (GOCO) facility that was operated by the Northrop Grumman Corporation (NGC) until September 1998. NWIRP Bethpage is bordered on the north, west, and south by property owned, or formerly owned, by NGC that covered approximately 605 acres, and, on the east, by a residential neighborhood.

The GM-38 Area refers to a cluster of monitoring wells that were installed in the 1990s by NGC and that first identified an isolated groundwater contaminant plume in this area. The GM-38 Area is approximately 8,500 feet south-southeast and hydraulically downgradient of NWIRP Bethpage. The GWTP is located within a utility easement that is located east of Broadway Avenue, west of the Seaford Oyster Bay Expressway (Route 135), and between the north and south dead ends of Windhorst and Herman Avenues.

10.1.2 Site Background

NWIRP Bethpage is currently listed by NYSDEC as an “inactive hazardous waste site” (#1-30-003B), as is NGC (#1-30-003A) and the Hooker/RUCO site (#1-30-004) located less than 1/2 mile west of NWIRP Bethpage.

NWIRP Bethpage was established in 1941. Since inception, the primary mission of the facility has been the research, prototyping, testing, design engineering, fabrication, and primary assembly of military aircraft. The facilities at NWIRP Bethpage include four plants (Nos. 3, 5, and 20, used for assembly and prototype testing; and No. 10, which contains a group of quality control laboratories), two warehouse complexes, a salvage storage area, water recharge basins, an industrial wastewater treatment plant, and several smaller support buildings.

Historical operations that resulted in hazardous material generation at the facility included metal finishing processes, maintenance operations, painting of aircraft and components, and other activities that involve aircraft manufacturing. Wastes generated by plant operations were disposed directly into either drainage sumps, dry wells, and/or on the ground surface, resulting in the disposal of a number of hazardous wastes, including the volatile organic compounds (VOCs) tetrachloroethene (PCE) and trichloroethene (TCE), the semivolatile organic compounds (SVOCs) polychlorinated biphenyls (PCBs), and the inorganic analytes chromium and cadmium. Some of these contaminants have migrated from the points of disposal to surrounding areas, including the soils of these surrounding areas and the groundwater beneath and downgradient of the NWIRP Bethpage property.

Chlorinated VOCs were identified in the GM-38 Area in moderately deep (220 to 470 feet below ground surface [bgs]) groundwater at concentrations greater than 500 micrograms per liter (µg/L). The contaminated groundwater in the area represents a relatively large mass of chlorinated VOCs that would remain for extended periods and could adversely affect public water supplies in the area, as well as other downgradient water supplies. Two public water supply systems are present in the general area and extract groundwater at depths ranging from 540 to 740 feet bgs. Navy- and contractor-funded systems are in place at the public water supply wells to remove VOCs from the water prior to distribution.

10.2 PROBLEM SUMMARY

As stated in the Navy's Record of Decision (ROD), the purpose of the groundwater treatment system is to "Eliminate, to the extent practical, site-related contaminants from the affected public water supplies and to prevent, to the extent practical, the future contamination of public water supplies through the implementation of the offsite groundwater remediation." The treatment system has been designed to pump groundwater from two recovery wells RW-1 and RW-3 at a rate of 1,100 gpm and discharge into an injection well or recharge basin after removal of targeted chlorinated VOCs as per the requirements of the SPDES permit equivalent (1,1-dichloroethane, 1,2-dichloroethane, 1,1-dichloroethene, cis-1,2-dichloroethene, trans-1,2-dichloroethene, PCE, 1,1,1-trichloroethane, TCE, and vinyl chloride).

The treatment consists of air stripping followed by polishing with liquid-phase granular activated carbon (LGAC). The stripped vapors are treated in the vapor-phase granular activated carbon (VGAC) unit as per the NYSDEC air emissions requirements before being discharged into the atmosphere via the exhaust stack. Mercury has been listed in the SPDES requirements as a parameter to be monitored. The treatment system has not been designed to remove mercury, although low levels of mercury that are present in the regional groundwater could be removed by the LGAC unit.

The capture zone for the groundwater is determined by the drawdown induced by the pumping of recovery wells RW-1 and RW-3 at 1,100 gpm. The treatment system has been designed for a 5 to 10 year operational life. It is not intended to remediate groundwater contamination in the local aquifer to non-detectable levels. Rather, the intent of the system is to remove mass, reduce elevated VOC levels to levels similar to those in the surrounding aquifer, and minimize the impacts on water supply wells and currently unaffected portions of the aquifer.

Please refer to the OM&M Plan for further details.

10.3 PROJECT DECISION STATEMENTS

This SAP was prepared primarily to address samples to be collected from the GWTP and the eight monitoring wells and used to evaluate the effectiveness of the treatment plant.

1. If weekly grab samples from the effluent show no exceedances of the stated discharge limitations for 24 consecutive weekly sampling events then, the sampling will be monthly.

2. Monthly grab samples will be collected from the influent, effluent, and two intermediate locations of the vapor phase treatment system. If the results of these monthly grab samples show exceedances of allowable limits for the air permit equivalent then, the GWTP vapor phase treatment system may need to be modified.

3. Water level measurements will be performed in all 12 monitoring wells on a quarterly basis. In addition, samples for water quality monitoring will be collected from eight of the 12 wells on a quarterly basis for the first two years from the start of GWTP operations, on a semi-annual basis for years three and four, and then on an annual basis from the fifth year onwards.

    - If the water level measurements do not show a sufficient capture zone for the groundwater "hot spot" (with the capture zone being determined by observed drawdown in the recovery wells and adjacent monitoring wells when compared with baseline water levels prior to pumping), then the placement, the screened intervals, and/or the pumping rates from the recovery wells may need to be modified.

- If the groundwater sample results from the eight wells show exceedances of Project Action Limits (PALs; see Worksheet #15), then, depending on concentration and/or occurrence, the GWTP operations may need to be modified and/or the sampling frequency may not be reduced during the plant operations.
- If groundwater sample results show concentrations of chlorinated VOCs in the GM-38 Area groundwater "hot spot" equal to concentrations in the surrounding aquifer, then GWTP operations may be terminated.
- If groundwater sample results show concentrations of chlorinated VOCs in the GM-38 Area groundwater "hot spot" increasing after the operations at the GWTP are terminated (i.e., during post-shut down monitoring), then the operations may be resumed.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

Project quality objectives (PQOs) are qualitative and quantitative statements that specify the type, quantity, and quality of the data required to support decisions during remedial activities. PQOs can be defined as what the end user expects to obtain from the analysis results, and are developed by the entire project team using a systematic planning process. Figure 11-1 presents a flowchart of this process; although the activities are presented in a sequential manner, the entire planning process is iterative and earlier activities can, and should, be re-visited as required throughout the life of the project.

PQOs are generally developed through a seven step process, with a graded approach being implemented according to the complexity of the project:

- Step 1 State the problem
- Step 2 Identify the decision
- Step 3 Identify inputs to the decision
- Step 4 Define the study boundaries
- Step 5 Develop a decision rule
- Step 6 Specify limits on decision errors
- Step 7 Optimize the design for obtaining data

Worksheets #9 and #10 contain information on the project scoping sessions and project definition. Overall project objectives are:

- Sample and monitor influent and effluent streams during the start-up period and the prove-out period.
- Sample and monitor process streams (including influent and effluent) during routine operations.
- Monitor the groundwater level in 12 monitoring wells and sample 8 of these 12 monitoring wells.

Who Will Use the Data?
TtEC, NYSDEC (regulatory oversight), and Navy (property owner).

What Will the Data be Used For?
- To monitor the operations within the GWTP;
- To confirm the GWTP effluent is acceptable for discharge per the SPDES permit equivalent;
- To confirm the GWTP vapor is acceptable for discharge per the air permit equivalent; and
- To monitor hydraulic containment and capture of the groundwater “hot spot.”

What Type of Data are Needed?
The sampling program will include the following:

Sampling and Monitoring during the Start-Up – Sampling and monitoring of the GWTP performance will be implemented during the start-up period in 2009 in order to ensure that the system is operating properly and that effluent streams meet all regulatory, and disposal facility requirements. The start-up period is defined as the first 30 days of operations. During the start-up period, 12 process streams (either aqueous or air) will be sampled and/or monitored as shown on Worksheet #18. The purpose of this sampling and monitoring is for tracking and documenting GWTP operations performance as well as for regulatory compliance. During the system start-up period, all samples will be collected on a weekly basis. Only the process effluent streams will be sampled for regulatory compliance purposes for the parameters identified by NYSDEC and for disposal purposes for the parameters identified by the disposal facilities, at the designated frequency. All analytical parameters for regulatory compliance will analyzed at a laboratory certified by the State of New York for these parameters.

- Sampling and Monitoring during the Prove-Out – Sampling and monitoring of the GWTP performance will be implemented during the prove-out period in 2009 and 2010 in order to ensure that the GWTP is operating in accordance with the design specifications and that effluent streams meet all regulatory, and disposal facility requirements. The prove-out period is defined as the second through the sixth months of operations. During the prove-out period, the same 12 process streams (either aqueous or air) will be sampled and/or monitored (see Worksheet #18). The purpose of this sampling and monitoring is to ensure the GWTP is operating in accordance with the design specifications and that effluent streams meet all regulatory, and disposal facility requirements. During the system prove-out period, all samples will be collected on a weekly basis. Based on the data that is collected during the start-up and prove-out periods, after consultation with the Navy RPM, TtEC may decide to reduce the frequency of sampling and analyses for the influent process water and some of the intermediate process streams. Only the process effluent streams will be sampled for regulatory compliance purposes for the parameters identified by NYSDEC and for disposal purposes for the parameters identified by the disposal facilities, at the designated frequency. All analytical parameters for regulatory compliance will be analyzed at a laboratory certified by the State of New York for these parameters.

- Sampling and Monitoring during Routine Operations – Routine operations will commence at the end of the prove-out period (i.e., after the end of the first six months of operations). During routine operations, the same 12 process streams (either aqueous or air) will be sampled and/or monitored (see Worksheet #18). It should be noted that most of this sampling and monitoring is for the purpose of tracking and documenting the performance of plant operations and not for regulatory compliance reporting purposes. Only the process effluent streams will be sampled for regulatory compliance purposes for the parameters identified by NYSDEC and for disposal purposes for the parameters identified by the disposal facilities, at the designated frequency. Based on experience gained in operating the GWTP, following the start-up and prove-out periods, after consultation with the Navy RPM, TtEC may reduce the frequency of sampling and analyses for the influent process water and intermediate process streams. All analytical parameters for regulatory compliance will be collected monthly (samples for pH will be collected weekly) and analyzed at a laboratory certified by the State of New York for these parameters. Samples from recovery wells RW-1 and RW-3 and treated effluent will be collected once every two weeks.

- Sampling and Monitoring of the Groundwater – Sampling and monitoring of the groundwater from the existing monitoring wells will be performed throughout the period of operation of the GWTP and for two years beyond the shut-down of GWTP operations to determine the effectiveness of the remediation activities and monitor the hydraulic containment and capture of the groundwater “hot spot” by the recovery wells. Water level measurements will be performed in all 12 monitoring wells on a quarterly basis. In addition, samples for water quality monitoring will be collected from eight of the 12 wells on a quarterly basis for the first two years from the start of GWTP operations, on a semi-annual basis for years three and four, and then on an annual basis from the fifth year onwards. It was deemed unnecessary to collect samples from all 12 wells because some of them are in close proximity to each other and are therefore expected to have the same water quality.

  The effectiveness of the treatment system will be determined by making a comparison between the concentrations of chlorinated VOCs within the “hot spot” with corresponding levels in the surrounding aquifer. When concentrations of chlorinated VOCs in the GM-38 Area groundwater “hot spot” are equal to those concentrations in the surrounding aquifer over a sustained period of time, TtEC will make the recommendation to the Navy that operations at the GWTP be terminated. The final determination to take the GWTP off-line will be made by the Navy in consultation with NYSDEC.

With consent from the Navy and NYSDEC, sampling and monitoring of the groundwater quality for the same parameters will continue for two years (on a quarterly basis) beyond the shut-down of the GWTP operations. If the concentrations of the chlorinated VOCs in the GM-38 Area groundwater "hot spot" increase after the operations at the GWTP are terminated, the operations may have to be resumed. This will be done, if necessary, after consultation with the Navy and NYSDEC.

Worksheet #17 presents the wells that will be sampled for water quality monitoring.

How "Good" do the Data Need to be in Order to Support the Environmental Decision?
The overall QA/QC objective for the sampling activities is to provide data of known and documented quality through the use of developed and implemented procedures. Quality characteristics for data are determined by the evaluation of the precision, accuracy, representativeness, comparability, and completeness (PARCC) of the analytical results, and sensitivity and blank contamination elimination. Data quality objectives for each of these parameters are determined based on the level of data required. Specific QA/QC objectives for the definitive data are presented on Worksheet #12.

How much data are needed?
The field program for the GM-38 Area GWTP is presented in Worksheets #18, #19, and #20.

Where, When, and How Should the Data be Collected/Generated?
Worksheet #16 presents the project schedule.

Worksheet #17 presents the sampling program design and rationale. Worksheet #18 presents the sampling methods. Worksheet #21 provides the standard operating procedures (SOPs) that govern the various types of sampling.

Who Will Collect and Generate the Data?
Qualified personnel from either TtEC or the GWTP operating subcontractor will collect samples from the 13 process streams (includes intermediate process streams). TtEC personnel or the GWTP operating subcontractor will collect samples from the monitoring wells. Personnel from the subcontracted laboratory will generate definitive data using specific analytical methods and guidelines. Additional information on project personnel is provided in Worksheets #5 through #7.

How Will the Data be Reported?
The analytical laboratories will tabulate and compile analytical results and associated QA/QC information. Results from the off-site laboratory analyses are to be reported on standard EPA forms in standard units for the matrix and analysis (e.g., ug/L for organics and metals in groundwater samples). Field screening parameters (e.g., pH) will be reported in standard units for the analysis. Tabular data will be provided in Excel files by the laboratory.

How will the Data be Archived?

- Data from TtEC's subcontract laboratories will be received in electronic and hardcopy formats specified in the contract and then reviewed by TtEC personnel.
- All electronic data will be input into the project's database.
- Generated data (field- and/or laboratory-related) will be stored in the project files when not undergoing processing/review.
- Hard copies of field data (i.e., field logbooks and field data sheets) will be archived in the project files.
- Hard copies of analytical data received by TtEC will be archived in the project files for 10 years after contract expiration.

SAP Worksheet #12 -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – *Aqueous Field Quality Control Samples*

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Cooler Temperature Indicator	VOCs, Mercury, and Total Suspended Solids (TSS)	One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees Celcius.	S
Field Duplicate		One per 20 samples	Precision (field)	50% Relative Percent Difference (RPD)	S&A
Field Blank*		One per 20 samples	Representativeness	No analyte > quantitation limit (QL)	S&A
All samples		All samples	Sensitivity	Method detection limits (MDLs) < project action limits listed on Worksheet #15	A
All samples		All samples	Data Completeness	95% overall	S&A
Trip Blank	VOCs	One per cooler	Accuracy	No analyte > QL	S&A

* - Applicable to groundwater sampling from the eight monitoring wells.

Measurement Performance Criteria Table – *Air Field Quality Control Samples*

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Duplicate	VOCs	1 per 20 samples	Precision (field)	50% RPD	S&A
All samples		All samples	Sensitivity	MDLs < project action limits listed on Worksheet #15	A
All samples		All samples	Data Completeness	95% overall	S&A

Measurement Performance Criteria Table – *Spent Air Stripper Media Field Quality Control Samples*

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Cooler Temperature Indicator	TCLP VOCs and TCLP Metals	One per cooler	Accuracy / Representativeness	Between 2 and 6 degrees Celcius.	S
All samples		All samples	Sensitivity	MDLs < project action limits listed on Worksheet #15	A
All samples		All samples	Data Completeness	95% overall	S&A

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Final Design	Final Design for GM-38 Area Groundwater Remediation, NWIRP Bethpage, NY, May 8, 2006	TtEC Design information for the GWTP	Construct and operate the GWTP.	None.
Record of Decision by Engineering Facility Northeast (EFANE)	Record of Decision NWIRP Bethpage, NY, Operable Unit 2 Groundwater, NYS Registry 1-3-003B, April 2003 (Revision 1)	EFANE Record of Decision	Identify remedial actions for groundwater at NWIRP.	None.
Record of Decision by NYSDEC	Record of Decision, Operable Unit 2 Groundwater, Northup Grumman and NWIRP Sites, Nassau County, Site Numbers 1-30-003A & B, March 2001	NYSDEC Record of Decision	Identify remedial actions for groundwater at Northup Grumman and NWIRP.	None.

SAP Worksheet #14 -- Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

Characterization activities for this project will include the following:

- Sampling and monitoring of the GWTP performance during the start-up period in 2009 in order to ensure that the system is operating properly and that effluent streams meet all regulatory and disposal facility requirements.

- Sampling and monitoring of the GWTP during the prove-out period in 2009 and 2010 in order to ensure that the system is operating in accordance with the design specifications and meets all regulatory and disposal facility requirements.

- Sampling and monitoring of influent, effluent, and intermediate process streams within the GWTP for the purpose of evaluating the operation and performance of the process equipment used for air stripping, bag filtration, liquid-phase and vapor-phase granular activated carbon adsorption, and backwashing during routine operations.

- Sampling and monitoring of the groundwater to determine the effectiveness of the remediation activities and monitor the hydraulic containment and capture of the groundwater "hot spot" by the recovery wells.

The SOPs and field forms referenced below and in the worksheets are included in Appendices A and B, respectively.

- **QC Tasks –** Worksheet #20 provides a summary of the required field QC samples by matrix. Field QC samples will be labeled and shipped according to the procedures outlined below.

  Field Equipment Rinsate Blanks
  A field blank will be collected to evaluate the potential for residual chemical contamination of environmental samples from inadequate decontamination of field equipment. Field blanks will be collected by pouring reagent (analyte-free) water over and/or through decontaminated equipment, and collecting the rinsate. Field blanks will be collected at a frequency of five percent of the total samples (i.e., one blank for up to every 20 samples) for chemical parameters when non-disposable sampling equipment is used. Analysis of field blanks will be identical to analysis of the associated environmental samples, and the blanks will be preserved as indicated in the applicable methods. Sufficient equipment will be available and field activities scheduled, as possible, to minimize the number of field blanks required.

  Trip Blanks
  A trip blank serves to detect possible cross-contamination of samples resulting from handling, storage and shipment procedures. Trip blanks consist of volatile organic analysis (VOA) vials filled with deionized (DI) water prior to initiation of daily field activities and preserved accordingly, which accompany the day's environmental samples through collection and shipment to the laboratory. In addition, trip blanks are stored by the laboratory under the same conditions as the environmental samples. A trip blank must accompany each cooler containing aqueous samples for VOC analysis, and will be analyzed identically to the associated environmental samples. All aqueous VOC samples will be consolidated in one cooler for daily shipment, as possible, to minimize the number of trip blanks required.

  Cooler Temperature Blanks
  A cooler temperature blank will be included in each cooler of samples shipped from the Site to verify that the cooler temperature has been maintained at 4 ± 2°C. A vial will be filled with either

potable or DI water, unpreserved, and labeled with "Cooler Temperature Indicator" and the date. The laboratory will record the temperature of the blank water on the chain of custody (COC) form immediately upon cooler arrival.

- **Analytical Tasks -** Analyses will be performed in accordance with the analytical methods identified in Worksheet #23. The subcontracted laboratories will meet the QLs specified in Worksheet #15. The subcontracted laboratories will perform the chemical analyses following laboratory-specific operating procedures developed based on the methods listed in Worksheet #23. The subcontracted laboratories will need to be accredited by DoD ELAP and maintain this approval throughout the project. See Appendices C and D for laboratory documentation.

  - The aqueous samples from the GWTP will be analyzed for the list of constituents outlined in the SPDES permit, i.e., select VOCs (1,1-dichloroethane, 1,2-dichloroethane, 1,1-dichloroethene, cis-1,2-dichloroethene, trans-1,2-dichloroethene, PCE, 1,1,1-trichloroethane, TCE, and vinyl chloride) and mercury, and/or total suspended solids (TSS) by a subcontracted laboratory, Test America – Pittsburgh. In addition, aqueous samples from the GWTP will be analyzed for pH with a hand-held pH meter.
  - The aqueous samples from the monitoring wells will be analyzed for Target Compound List (TCL) VOCs plus Tentatively Identified Compounds (TICs), mercury, and TSS by a subcontracted laboratory, Test America – Pittsburgh. In addition, the groundwater samples from the monitoring wells will be analyzed for pH just prior to sampling with a water quality meter. The full TCL VOC list was selected in order to compare the results with historical sampling data from the surrounding aquifer as part of the overall evaluation of remedial activities, and use of the same compound list and methodology increases data comparability. If assessment of the monitoring well groundwater data indicates re-occurring compound(s), the constituent(s) may be added to the laboratory analysis of the influent and/or effluent samples (first bullet above), after discussion and approval by the Navy and NYSDEC.
  - The air samples from the GWTP vapor phase will be analyzed for select VOCs (1,2-dichloroethane, 1,2-dichloroethene, toluene, 1,1,2-trichloroethane, TCE, vinyl chloride, and xylene) by a subcontracted laboratory, AirToxics, Ltd.
  - Spent air stripper media will be analyzed for Toxicity Characteristic Leaching Procedure (TCLP) VOCs and TCLP metals by a subcontracted laboratory, Test America – Pittsburgh, prior to disposal.

- **Data Management -** Project data will be managed according to the procedures outlined on the following worksheets:

  - Project documentation and records
    - Field sample collection and field measurement records - See Worksheets #27 and #29
    - Laboratory data package deliverables - See Worksheet #30
    - Data assessment documents and records - See Worksheet #29
  - Data recording formats - See Worksheet #27
  - Data handling, management, tracking, and control - See Worksheet #29

- **Assessment and Oversight** - See Worksheet #32 for assessment findings and corrective actions and Worksheet #33 for QA management reports.

- **Data Review -** Data reviews will be conducted in accordance with the procedures outlined on the following worksheets:

  - Data verification – See Worksheet #34
  - Data validation – See Worksheets #35 and #36
  - Usability assessment – See Worksheet #37

SAP Worksheet #15 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: *Aqueous*
Analytical Group: *VOCs*

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal[1] (µg/L)	Achievable Laboratory Limits[2]	
					QL (µg/L)	MDL (µg/L)
Dichlorodifluoromethane	75-71-8	5	NYSDEC[3]	1.0	1.0	0.2590
Chloromethane	74-87-3	5	NYSDEC[3]	1.0	1.0	0.2665
Vinyl Chloride	75-01-4	2	SPDES Effluent Limitation[4]	1.0	1.0	0.2906
Bromomethane	74-83-9	5	NYSDEC[3]	1.0	1.0	0.3039
Chloroethane	75-00-3	5	NYSDEC[3]	1.0	1.0	0.2491
Trichlorofluoromethane	75-69-4	5	NYSDEC[3]	1.0	1.0	0.0804
1,1-Dichloroethene	75-35-4	5	SPDES Effluent Limitation[4]	1.0	1.0	0.2814
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5	NYSDEC[3]	1.0	1.0	0.3016
Acetone	67-64-1	50	NYSDEC[3]	5.0	5.0	2.5
Carbon Disulfide	75-15-0	60	NYSDEC[3]	1.0	1.0	0.1956
Methyl Acetate	79-20-9	--	NYSDEC[3]	1.0	1.0	0.1025
Methylene Chloride	75-09-2	5	NYSDEC[3]	1.0	1.0	0.3177
trans-1,2-Dichloroethene	156-60-5	5	SPDES Effluent Limitation[4]	1.0	1.0	0.2722
Methyl tert-Butyl Ether	1634-04-4	10	NYSDEC[3]	1.0	1.0	0.2019
1,1-Dichloroethane	75-34-3	5	SPDES Effluent Limitation[4]	1.0	1.0	0.2432
cis-1,2-Dichloroethene	156-59-2	5	SPDES Effluent Limitation[4]	1.0	1.0	0.2738
2-Butanone	78-93-3	50	NYSDEC[3]	5.0	5.0	0.4981

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal[1] (µg/L)	Achievable Laboratory Limits[2]	
					QL (µg/L)	MDL (µg/L)
Bromochloromethane	74-97-5	5	NYSDEC[3]	1.0	1.0	0.2903
Chloroform	67-66-3	7	NYSDEC[3]	1.0	1.0	0.2415
1,1,1-Trichloroethane	71-55-6	5	SPDES Effluent Limitation[4]	1.0	1.0	0.2505
Cyclohexane	110-82-7	--	NYSDEC[3]	1.0	1.0	0.2972
Carbon Tetrachloride	56-23-5	5	NYSDEC[3]	1.0	1.0	0.3041
Benzene	71-43-2	1	NYSDEC[3]	1.0	1.0	0.2732
1,2-Dichloroethane	107-06-2	0.6	SPDES Effluent Limitation[4]	1.0	1.0	0.2145
Trichloroethene	79-01-6	5	SPDES Effluent Limitation[4]	1.0	1.0	0.2932
Methylcyclohexane	108-87-2	--	NYSDEC[3]	1.0	1.0	0.3165
1,2-Dichloropropane	78-87-5	1	NYSDEC[3]	1.0	1.0	0.1798
Bromodichloromethane	75-27-4	50	NYSDEC[3]	1.0	1.0	0.2016
cis-1,3-Dichloropropene	10061-01-5	0.4	NYSDEC[3]	1.0	1.0	0.1945
4-Methyl-2-Pentanone	108-10-1	--	NYSDEC[3]	5.0	5.0	0.2256
Toluene	108-88-3	5	NYSDEC[3]	1.0	1.0	0.2336
trans-1,3-Dichloropropene	10061-02-6	0.4	NYSDEC[3]	1.0	1.0	0.1834
1,1,2-Trichloroethane	79-00-5	1	NYSDEC[3]	1.0	1.0	0.1959
Tetrachloroethene	127-18-4	5	SPDES Effluent Limitation[4]	1.0	1.0	0.2361
2-Hexanone	591-78-6	50	NYSDEC[3]	5.0	5.0	0.5345
Dibromochloromethane	124-48-1	50	NYSDEC[3]	1.0	1.0	0.1649
1,2-Dibromoethane	106-93-4	--	NYSDEC[3]	1.0	1.0	0.2299
Chlorobenzene	108-90-7	5	NYSDEC[3]	1.0	1.0	0.2255

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal[1] (µg/L)	Achievable Laboratory Limits[2]	
					QL (µg/L)	MDL (µg/L)
Ethylbenzene	100-41-4	5	NYSDEC[3]	1.0	1.0	0.1816
Xylenes (total)	1330-20-7	5	NYSDEC[3]	3.0	3.0	0.6187
Styrene	100-42-5	5	NYSDEC[3]	1.0	1.0	0.2177
Bromoform	75-25-2	50	NYSDEC[3]	1.0	1.0	0.2453
Isopropylbenzene	98-82-8	5	NYSDEC[3]	1.0	1.0	0.2227
1,1,2,2-Tetrachloroethane	79-34-5	5	NYSDEC[3]	1.0	1.0	0.1539
1,3-Dichlorobenzene	541-73-1	3	NYSDEC[3]	1.0	1.0	0.1630
1,4-Dichlorobenzene	106-46-7	3	NYSDEC[3]	1.0	1.0	0.1780
1,2-Dichlorobenzene	95-50-1	3	NYSDEC[3]	1.0	1.0	0.2176
1,2-Dibromo-3-chloropropane	96-12-8	0.04	NYSDEC[3]	1.0	1.0	0.2411
1,2,4-Trichlorobenzene	120-82-1	5	NYSDEC[3]	1.0	1.0	0.1509
1,2,3-Trichlorobenzene	87-61-6	5	NYSDEC[3]	1.0	1.0	0.1587

BOLD identifies the select VOCs for which the GWTP aqueous samples will be analyzed.

1 The Project Quantitation Limit Goal may be higher for some constituents but the MDL is low enough to detect these compounds below the Project Action Limit as estimated concentrations.

2 Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

3 NYSDEC Ambient Water Quality Standards and Guidance Values, Class GA.

4 Based on NYSDEC Ambient Water Quality Standards and Guidance Values, Class GA.

Matrix: *Aqueous*

Analytical Group: *Mercury*

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Achievable Laboratory Limits[1]	
					QL (µg/L)	MDL (µg/L)
Mercury	7439-97-6	0.25	SPDES Effluent Limitation	0.2	0.2	0.0205

1 Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

Matrix: *Air*

Analytical Group: *VOCs*

Analyte	CAS Number	Project Action Limit[1] (lb/hr)	Project Action Limit Reference	Project Quantitation Limit Goal (ppbv	Achievable Laboratory Limits[2]	
					QL (ppbv)	MDL (pptv
1,2-Dichloroethane	107-06-2	0.09	NYSDEC Air Permit Emission Discharge Limit	0.5	0.5	78.68
1,2-Dichloroethene	540-59-0	0.01	NYSDEC Air Permit Emission Discharge Limit	0.5	0.5	127.18 (cis) / 226.14 (trans)
Toluene	108-88-3	0.03	NYSDEC Air Permit Emission Discharge Limit	0.5	0.5	65.90
1,1,2-Trichloroethane	79-00-5	Below Reporting Threshold	NYSDEC Air Permit Emission Discharge Limit	0.5	0.5	144.26
Trichloroethene	79-01-6	Below Reporting Threshold	NYSDEC Air Permit Emission Discharge Limit	0.5	0.5	80.34
Vinyl Chloride	75-01-4	Below Reporting Threshold	NYSDEC Air Permit Emission Discharge Limit	0.5	0.5	174.47
Xylene	1330-20-7	Below Reporting Threshold	NYSDEC Air Permit Emission Discharge Limit	0.5	0.5	92.71 (m,p) / 126.54 (o)

1 The project action level is presented in pounds per hour as it was presented in air permit equivalent approved by NYSDEC.
2 Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs are instrument specific and will be determined during sample analysis. The provided MDLs are from an example Standard Level (MSD-2) MDL Summary Report as provided by the laboratory (see Appendix D).

SAP Worksheet #16 -- Project Schedule / Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Activity	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Draft OM&M Plan/UFP-SAP to Navy	TtEC	07/15/09		Draft OM&M Plan/UFP-SAP	
Navy Review	Navy			NA	NA
Revised Draft OM&M Plan/UFP-SAP to Regulators	TtEC	NA	NA	Draft OM&M Plan/UFP-SAP	
Regulator Review	NYSDEC	NA	NA	NA	
Draft Final OM&M Plan/UFP-SAP to Regulators	TtEC	08/05/09	9/25/09	Draft Final OM&M Plan/UFP-SAP	09/25/09
Regulator Review	NYSDEC	9/25/09	10/25/09	NA	
Final OM&M Plan/UFP-SAP to Navy and Regulators	Navy and NYSDEC	7/10	09/17/10	Final OM&M Plan/UFP-SAP	11/25/09
Discharge Monthly Reports	TtEC	TBD	TBD	Discharge Monthly Reports	Monthly
Data Review	TtEC	TBD	TBD	Final Analytical Data Tables	TBD

SAP Worksheet #17 -- Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)

Several sampling and monitoring programs will be conducted as part of the GWTP operations. These include:

1) sampling and monitoring of influent, effluent, and intermediate process streams during the start-up period;
2) sampling and monitoring of influent, effluent, and intermediate process streams during the prove-out period;
3) sampling and monitoring of process streams (including influent, effluent, and intermediate) for routine operations; and
4) sampling and monitoring of groundwater.

17.1 SAMPLING AND MONITORING GWTP PERFORMANCE DURING START-UP PERIOD

Sampling and monitoring of the GWTP performance will be implemented during the start-up period in 2009 in order to ensure that the system is operating properly and that effluent streams meet all regulatory and disposal facility requirements. The start-up period is defined as the first 30 days of operations. During the start-up period, the process streams as listed on Worksheet #18 will be sampled and/or monitored. The purpose of this sampling and monitoring is for tracking and documenting GWTP operations performance as well as for regulatory compliance. During the system start-up period, all samples will be collected on a weekly basis. Only the process effluent streams will be sampled for regulatory compliance purposes for the parameters identified by NYSDEC and for disposal purposes for parameters as required by the disposal facilities, at the designated frequency. All analytical parameters for regulatory compliance will be collected weekly and analyzed at a laboratory certified by the State of New York for these parameters, Test America – Pittsburgh and Air Toxics, Ltd.

17.2 SAMPLING AND MONITORING GWTP PERFORMANCE DURING PROVE-OUT PERIOD

Sampling and monitoring of the GWTP performance will be implemented during the prove-out period in 2009 in order to ensure that the GWTP is operating in accordance with the design specifications and that effluent streams meet all regulatory and disposal facility requirements. The prove-out period is defined as the second through the sixth months of operations. During the prove-out period, the process streams as listed on Worksheet #18 will be sampled and/or monitored. The purpose of this sampling and monitoring is for ensuring that the GWTP is operating in accordance with the design specifications and that effluent streams meet all regulatory and disposal facility requirements. During the system prove-out period, all samples will be collected on a weekly basis. Based on the data that is collected during the start-up and prove-out periods, after consultation with the Navy RPM, TtEC may decide to reduce the frequency of sampling and analyses for the influent process water and some of the intermediate process streams. Only the process effluent streams will be sampled for regulatory compliance purposes for the parameters identified by NYSDEC and for disposal purposes for parameters as required by the disposal facilities, at the designated frequency. All analytical parameters for regulatory compliance will be collected weekly and analyzed at a laboratory certified by the State of New York for these parameters, Test America – Pittsburgh and Air Toxics, Ltd.

17.3 SAMPLING AND MONITORING FOR ROUTINE OPERATIONS

Routine operations will commence at the end of the prove-out period (i.e., after the end of the first six months of operations). During routine operations, the process streams as listed on Worksheet #18 will be sampled and/or monitored. It should be noted that most of this sampling and monitoring is for the purpose of tracking and documenting the performance of plant operations and not for regulatory compliance reporting purposes. Only the process effluent streams will be sampled for regulatory compliance purposes for the parameters identified by NYSDEC and for disposal purposes for parameters as required by the disposal facilities, at the designated frequency. Based on experience gained in

operating the GWTP following the start-up and prove-out periods, after consultation with the Navy RPM, TtEC may reduce the frequency of sampling and analyses for the influent process water and some of the intermediate process streams. All analytical parameters for regulatory compliance will be collected monthly (samples for pH will be collected weekly) and analyzed at a laboratory certified by the State of New York for these parameters, Test America – Pittsburgh and Air Toxics, Ltd. Samples from recovery wells RW-1 and RW-3 and treated effluent will be collected once every two weeks.

17.4 SAMPLING AND MONITORING OF GROUNDWATER IN GM-38 AREA

Sampling and monitoring of the groundwater from up to 12 monitoring wells will be performed throughout the period of operation of the GWTP and for two years beyond the shut-down of GWTP operations to determine the effectiveness of the remediation activities and monitor the hydraulic containment and capture of the groundwater "hot spot" by the recovery wells. The 12 wells are located as follows:

- There will be three monitoring wells near recovery well RW-1 that are screened between 395 and 435 feet below ground surface (feet bgs). RW-1 MW-1 is located approximately 140 feet northwest of RW-1, and RW-1 MW-2 is located approximately 50 feet north of RW-1. RW-1 MW-3 is proposed to be located approximately 400 feet northeast of RW-1, on the eastern side of Seaford Oyster Bay Expressway.

- There are three monitoring wells near recovery well RW-2 that are screened between 470 and 510 feet bgs. RW-2 MW-1 is located approximately 60 feet northwest of RW-2, RW-2 MW-2 is located approximately 20 feet west of RW-2, and RW-2 MW-3 is located approximately 100 feet west of RW-2.

- There are four proposed monitoring wells near recovery well RW-3. Two of these four wells (RW-3 MW-1 and RW-3 MW-3) are proposed to be screened between 320 and 340 feet bgs. The other two wells (RW-3 MW-2 and RW-3 MW-4) are proposed to be screened between 475 and 495 feet bgs. RW-3 MW-1 and RW-3 MW-2 are proposed to be located approximately 500 feet west of cluster GM-38, at the intersection of Arthur Avenue and Leroy Avenue. RW-3 MW-3 and RW-3 MW-4 are proposed to be located approximately 400 feet north of the intersection of Arthur Avenue and Broadway, on Broadway between Helena Avenue and Russell Avenue.

- There is one monitoring well near injection well IW-1. IW-1 MW-1 is screened between 130 and 150 feet bgs and is located approximately 20 feet south of IW-1.

- There are two monitoring wells GM-38D and GM-38D2 that are located approximately 320 feet west of RW-2, at the corner of Arthur Avenue and Broadway. GM-38D is screened between 320 and 340 feet bgs and GM-38D2 is screened between 475 and 495 feet bgs. TtEC does not have access to GM-38D and GM-38D2 for monitoring purposes.

- Monitoring well TP-1 is proposed to be screened between 450 and 470 feet bgs and to be located approximately 350 feet north of the GWTP building, alongside the GWTP access road.

Water level measurements will be performed in all 12 monitoring wells on a quarterly basis. In addition, samples for water quality monitoring will be collected from eight of the 12 wells (RW-1 MW-1, RW-1 MW-3, RW-2 MW-1, RW-3 MW-1, RW-3 MW-2, RW-3 MW-3, RW-3 MW-4, TP1) on a quarterly basis for the first two years from the start of GWTP operations, on a semi-annual basis for years three and four, and then on an annual basis from the fifth year onwards. It was deemed unnecessary to collect samples from all 12 wells because some of them are in close proximity and are therefore expected to have the same water quality. The final determination to take the GWTP off-line will be made by the Navy in consultation with NYSDEC. When concentrations of chlorinated VOCs in the GM-38 Area groundwater "hot spot" are equal to those concentrations in the surrounding aquifer, TtEC will make the recommendation to the Navy that operations at the GWTP be terminated. With consent from the Navy and NYSDEC, sampling and monitoring of the groundwater quality for the same parameters will continue for two years (on a quarterly basis) beyond the shut-down of the GWTP operations.

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling Location	Matrix	Analytical Group	Number of Samples	Sampling SOP Reference[1]
Influent from RW-1 – sample port BV-103	Aqueous	Select VOCs, Mercury, TSS, pH[2]	1 per sampling event	1
Influent from RW-3 – sample port BV-104	Aqueous	Select VOCs, Mercury, TSS, pH[2]	1 per sampling event	1
Air Stripper Effluent – sample port BV-115	Aqueous	Select VOCs, Mercury, TSS, pH[2]	1 per sampling event	1
Bag Filter Effluent – sample port BV-124	Aqueous	Select VOCs, Mercury, TSS, pH[2]	1 per sampling event	1
Treated Effluent to stormwater manhole and injection well IW-1 – sample port BV-127	Aqueous	Select VOCs, Mercury, pH[2]	1 per sampling event	1
LGAC-1 outlet (for series flow) – sample port BV-145	Aqueous	Select VOCs, Mercury, pH[2]	1 per sampling event	1
LGAC-2 outlet (for series flow) – sample port BV-149	Aqueous	Select VOCs, Mercury, pH[2]	1 per sampling event	1
LGAC-3 outlet (for series flow) – sample port BV-153	Aqueous	Select VOCs, Mercury, pH[2]	1 per sampling event	1
Off-gas VGAC-1 Inlet – sample port BV-132	Air	Select VOCs	1 per sampling event	2
Off-gas between VGAC-1 and VGAC-2 – sample port BV-134	Air	Select VOCs	1 per sampling event	2
Off-gas between VGAC-2 and VGAC-3 – sample port BV-136	Air	Select VOCs	1 per sampling event	2
Off-gas between VGAC-3 and Exhaust Stack – sample port BV-139	Air	Select VOCs	1 per sampling event	2
Monitoring Wells	Aqueous	TCL VOCs, Mercury, TSS, pH[2]	8 during each round[3]	3, 4, 5
Spent air stripper packing /filter / adsorber media – composite sample	Spent Air Stripper Media	TCLP VOCs, TCLP Metals	TBD[4]	8

1 SOP or worksheet that describes the sample collection procedures. SOPs are provided in Appendix A.

2 A hand-held meter will be used to analyze for pH.

3 All 12 wells will have groundwater level measurements collected prior to each round of groundwater sampling.

4 The number of samples will be determined during the initial operation of the GWTP. It is unknown at this time how often the media will need to be replaced.

SAP Worksheet #19 -- Analytical SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	**Analytical Group**	**Analytical and Preparation Method / SOP Reference**[1]	**Containers** (number, size, and type)	**Sample volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time**[2] (preparation / analysis)
Aqueous	VOCs (Select and TCL)	EPA 624	(3) 40 ml VOA vials w/Teflon lined septum	5 ml	1:1 HCl to pH<2; cool to 4°C	14 days
Aqueous	Mercury	EPA 245.1	1 500-mL HDPE	100 ml	1:1 HNO_3 to pH<2; cool to 4°C	28 days
Aqueous	TSS	SM 2540D	1 250-mL HDPE	250 ml	cool to 4°C	7 days
Spent Air Stripper Packing/ Filter /Adsorber Media	TCLP VOCs	SW-846 1311/8260B	(3) 40 ml VOA vials w/Teflon lined septum or 1 4-oz glass jar with Teflon lined septum	100 grams	cool to 4°C	14 days / 14 days
Spent Air Stripper Packing/ Filter /Adsorber Media	TCLP Metals	SW-846 1311/6010C/7470A	1 8-oz glass jar	100 grams	cool to 4°C	180 days / 180 days (except Mercury – 28 days / 28 days)
Trip Blank	VOCs (Select and TCL)	EPA 624	(2) 40 ml VOA vials w/Teflon lined septum	5 ml	1:1 HCl to pH<2; cool to 4°C	14 days
Air	Select VOCs	TO-15	1 6-liter Summa canister	1 L	None	30 days

1 See the Analytical SOP References Table (Worksheet #23).
2 Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Trip blanks will be collected when aqueous samples for VOC analysis are being collected. A trip blank serves to detect possible cross-contamination of samples resulting from handling, storage and shipment procedures. Trip blanks consist of VOA vials filled with DI water prior to initiation of daily field activities and preserved accordingly, which accompany the day's environmental samples through collection and shipment to the laboratory. In addition, trip blanks are stored by the laboratory under the same conditions as the environmental samples. A trip blank must accompany each cooler containing aqueous samples for VOC analysis, and will be analyzed identically to the associated environmental samples. All VOC samples will be consolidated in one cooler for daily shipment, as possible, to minimize the number of trip blanks required for the field program.

Field rinsate blanks will be collected during groundwater sampling of the monitoring wells. Laboratory supplied field blank water will be pumped through a decontaminated submersible pump into VOA vials for VOC analysis and into high density polyethylene (HDPE) bottles for mercury. Field rinsate blanks will be collected one per decontamination event, not to exceed 1 per 20 samples.

Field duplicate samples will be collected during groundwater sampling of the monitoring wells, process water sampling, and air sampling from the stack of the vapor-phase granular activated carbon adsorption units. Field duplicates will be collected one per 20 samples per media. Field duplicates will be collected in the same manner for the same analyses as the primary sample for which it is a duplicate.

All aqueous samples designated for chemical analysis will be put in coolers with ice to cool the samples to 4º C. Temperature blanks will be added to each cooler to verify that samples were stored at the correct temperature during shipment. These blanks will be used to help qualitatively determine the representativeness and comparability of the data as well as any biases or imprecision that might result from deviations in storage requirements. Elevated storage temperatures can cause loss of target analytes (e.g., via microbial degradation). The air samples will be shipped in boxes to the laboratory as cooling is not required.

Matrix	Analytical Group	No. of Sampling Locations[1]	No. of Field Duplicates	No. of MS/MSD[2]	No. of Field Blanks	No. of VOA Trip Blanks[3]	No. of PT Samples[4]	Total No. of Samples to Lab
Process Water - Aqueous	Select VOCs	8	1	1	0	1	0	11
Process Water - Aqueous	Mercury	8	1	1	0	0	0	10
Process Water - Aqueous	TSS	8	1	0	0	0	0	9
Process Water - Aqueous	pH[5]	8	0	0	0	0	0	8
Process Air	Select VOCs	4	1	0	0	0	0	5
Spent Air Stripper Packing/ Filter /Adsorber Media	TCLP VOCs	TBD[6]	0	0	0	0	0	TBD[5]
Spent Air Stripper Packing/ Filter /Adsorber Media	TCLP Metals	TBD[6]	0	0	0	0	0	TBD[5]
Groundwater - Aqueous	TCL VOCs	8	1	1	1	2	0	13
Groundwater - Aqueous	Mercury	8	1	1	1	0	0	11
Groundwater - Aqueous	TSS	8	1	0	0	0	0	9
Groundwater - Aqueous	pH[5]	8	0	0	0	0	0	8

1 Samples will be collected at the same location for multiple rounds. The numbers presented in this table are for one round of process aqueous and air sampling and one round of groundwater sampling.

2 Although the matrix spike/matrix spike duplicate (MS/MSD) is not typically considered a field QC sample, it is included here because location determination is often established in the field.
3 One per cooler per day of sampling for VOCs.
4 Batch or project-specific proficiency testing (PT) samples is optional. PT samples require additional field sample for analyses.
5 A hand-held meter will be used to analyze for pH.
6 The number of samples will be determined during the initial operation of the GWTP. It is unknown at this time how often the media will need to be replaced.

SAP Worksheet #21 -- Project Sampling SOP References Table
(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
1	Sampling of Process Aqueous Samples, Rev 0	TtEC	NA	N	
2	Sampling of Process Vapor Samples, Rev 0	TtEC	NA	N	
3	Water Level Measurement, Rev 0	TtEC	Water Level Indicator	N	
4	Field Parameter Measurements During Groundwater Sampling, Rev 0	TtEC	Water Quality Meter	N	
5	Groundwater Sampling [Low Flow Purge Procedure], Rev 0	TtEC	Low Flow Submersible Pump	N	
6	Decontamination – Field Instrumentation – Probes, Water Quality Meters, etc., Rev 0	TtEC	NA	N	
7	Decontamination – Non-disposable Chemical Sampling Equipment, Rev 0	TtEC	NA	N	
8	Sampling of Spent Air Stripper Packing/Filter/Adsorber Material, Rev 0	TtEC	NA	N	

SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Calibration Activity	Maintenance Activity	Testing/ Inspection Activity	Frequency	Acceptance Criteria		Corrective Action	Responsible Person	SOP Reference
PID	Calibrate with standard gasses	NA	NA	Prior to day's activities; end of day's activities; anytime anomaly suspected	+/- 5 ppm		Clean probe, replace battery, replace probe	TtEC FOL	SOPs 3, 5
PID	NA	NA	Visual inspection	Prior to day's activities	No defects noted		Replace probe	TtEC FOL	SOPs 3, 5
PID	NA	Check/replace battery	NA	Prior to day's activities; anytime anomaly suspected	+/- 5 ppm		Replace battery; replace probe	TtEC FOL	SOPs 3, 5
Water Level Indicator	NA	NA	Visual inspection	Prior to day's activities; end of day's activities; anytime anomaly suspected	No defects noted		Replace	TtEC FOL	SOPs 3, 5, 6
Water Level Indicator	NA	NA	Auditory inspection	Prior to day's activities; end of day's activities; anytime anomaly suspected	Audio tone for contact with water (markings in increments of ± 0.01 feet)		Replace	TtEC FOL	SOPs 3, 5, 6
Water Level Indicator	NA	Check/replace battery	NA	Prior to day's activities; anytime anomaly suspected	Audio tone for contact with water/NAPL		Replace battery	TtEC FOL	SOPs 3, 5, 6
Water Quality Meter	Calibrate with standard solutions	NA	NA	Prior to day's activities; end of day's activities; anytime anomaly suspected	pH Meter	± 0.1 units	Clean probe, replace battery, replace membrane, replace probe	TtEC FOL	SOPs 4, 5, 6
					Dissolved Oxygen	± 3%			
					Specific Conductivity	± 1% of full scale			
					ORP	± 10 mV			
					Temperature	± 0.1 °C			
					Turbidity	± 2 NTU			

Field Equipment	Calibration Activity	Maintenance Activity	Testing/ Inspection Activity	Frequency	Acceptance Criteria		Corrective Action	Responsible Person	SOP Reference
Water Quality Meter	NA	NA	Visual inspection	Prior to day's activities	No defects noted		Replace probe	TtEC FOL	SOPs 4, 5, 6
Water Quality Meter	NA	Check/replace battery	NA	Prior to day's activities; anytime anomaly suspected	pH Meter	± 0.1 units	Replace battery; replace probe	TtEC FOL	SOPs 4, 5, 6
					Dissolved Oxygen	± 3%			
					Specific Conductivity	± 1% of full scale			
					ORP	± 10 mV			
					Temperature	± 0.1 °C			
					Turbidity	± 2 NTU			

SAP Worksheet #23 -- Analytical SOP References Table
(UFP-QAPP Manual Section 3.2.1)

SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
EPA 624	Appendix A to Part 136, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 624 - Purgeables	Definitive	Aqueous – VOCs (Select and TCL)	Gas Chromatograph / Mass Spectrometer (GC/MS)	Test America – Pittsburgh	N
EPA 245.1	Method 245.1, Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry, Revision 3.0	Definitive	Aqueous – Mercury	Cold Vapor Atomic Absorption (AA)	Test America – Pittsburgh	N
SM 2540D	Solids in Water	Screening	Aqueous – TSS	Analytical Balance	Test America – Pittsburgh	N
SW-846 1311/8260B	Toxicity Characteristic Leaching Procedure / Volatile Organic Compounds by GC/MS	Definitive	Spent Air Stripper Media – TCLP VOCs	GC/MS	Test America – Pittsburgh	N
SW-846 1311/6010C/ 7470A	Toxicity Characteristic Leaching Procedure / Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) / Mercury in Liquid Waste (Manual Cold-Vapor Technique)	Definitive	Spent Air Stripper Media – TCLP Metals	ICP-AES / Cold Vapor AA	Test America – Pittsburgh	N
TO-15	Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By GC/MS	Definitive	Air – Select VOCs	GC/MS	Air Toxics, Ltd.	N

1 If yes, then specify the modification that has been made. Note that any analytical SOP modification made relative to project specific needs must be reviewed and approved by the Navy Quality Assurance Officer (QAO).

SAP Worksheet #24 -- Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS	BFB Tuning	Prior to ICAL and every 12 hours	Table 2 of EPA 624	Correct problem, then re-tune	Subcontract Laboratory GC/MS Technician	EPA 624
	Initial Calibration (ICAL)	After major instrument maintenance; prior to sample analysis	Relative Standard Deviation (RSD) < 35%	Check instrument performance, perform maintenance, recalibrate		
	QC Check Sample / Continuing Calibration Verification (CCV)	Daily (every 12 hours)	Table 5 of EPA 624	Check instrument performance, perform maintenance/ correct problem, recalibrate; retest		
GC/MS	BFB Tuning	Every 24 hours	Table 4 of SW-846 Method 8260; CCV internal standard area counts compared to ICAL with corrective action for > 40%D	Correct problem, then re-tune	Subcontract Laboratory GC/MS Technician	TO-15
	Initial Calibration (5-Point)	Prior to sample analysis	RSD ≤ 30% with two compounds allowed out to ≤ 40%	Correct problem, then repeat initial calibration		

GC/MS (cont'd)	Continuing Calibration (CCV)	At the start of each day and/or every 12 hours	70 – 130% (Under no circumstances will samples be analyzed if any compound exceeds 60-140%)	If up to two compounds exceed, flag associated data. If more than two, perform maintenance and repeat test. If systems still fails, perform new 5-point calibration curve.		TO-15 (cont'd)
Cold Vapor AA	Initial Calibration Instrument performance check (IPC) solution	Every analytical run Beginning of run, every 10 samples, end of run	correlation coefficient >=0.995 Analysis of the IPC solution immediately following calibration must verify that the instrument is within ±5% of calibration. Subsequent analyses of the IPC solution must be within ±10 % of calibration	Discontinue analysis, determine cause and/or in the case of drift the instrument recalibrated; reanalyze all samples following the last acceptable IPC	Subcontract Laboratory Cold Vapor Technician	EPA 245.1

Cold Vapor AA (cont'd)	Quality control sample (QCS)	Quarterly basis, after the preparation of stock or calibration standard solutions or as required to meet data-quality needs	±10% of true value	Identify source of problem, must be corrected before either proceeding on with the initial determination of method detection limits or continuing with ongoing analyses		EPA 245.1 (cont'd)
	Linear dynamic range (LDR)	Annually or whenever change in analytical performance	±10% of stated value of standard	Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed		

1 See Analytical SOP References Table (Worksheet #23).

2 Waste disposal (spent air stripper media) analyses are not included on the table.

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner, replace Electron Mulitplier	Monitor instrument performance via QC Check Sample/CCV	Monitor instrument performance via QC Check Sample/CCV	Daily, after every 12 hours of operation	%Difference < = 30%	Replace connections, replace gas line filters, replace trap, replace GC column, clip column, replace injection port liner, clean injection port, replace Electron Multiplier	Subcontract Laboratory GC/MS Technician	EPA 624
GC/MS	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner, replace Electron Mulitplier	Monitor instrument performance via CCV	Monitor instrument performance via CCV	Daily, after every 12 hours of operation	70 – 130%	Replace connections, replace gas line filters, replace trap, replace GC column, clip column, replace injection port liner, clean injection port, replace Electron Multiplier	Subcontract Laboratory GC/MS Technician	TO-15
Cold Vapor AA	Perform leak test, change tubing, clean window, clean filters	Monitor instrument performance via IPC	Monitor instrument performance via IPC	Daily, after every 10 samples, and at end of run	within +/- 10%, Hg not > QL	Replace connections, replace pump tubing, clean all filters	Subcontract Laboratory Cold Vapor Technician	EPA 245.1

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Analytical Balance	Clean surface	Weigh known National Institute of Standards and Technology (NIST) calibrated weights	Class "S" weights Class "1" Weights	Daily when in use Monthly	Criteria dependent on type of balance	Recalibration	Subcontract Laboratory Technician	All that involve any weighing of samples
Thermometers	NA	Calibration	Calibration against NIST thermometer	Annual	Temperature within ± 1 scale division	Replace thermometer if unable to calibrate	Subcontract Laboratory Technician	All sample/ standard storage units and drying ovens use thermometers

1 See Analytical SOP References Table (Worksheet #23).

2 Waste disposal (spent air stripper media) analyses are not included on the table.

SAP Worksheet #26 – Sample Handling System
(UFP-QAPP Manual Appendix A)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL (TBD)/TtEC or Plant Operator (Fred Mattison)/ECOR Solutions, Inc.
Sample Packaging (Personnel/Organization): FOL (TBD)/TtEC or Plant Operator (Fred Mattison)/ECOR Solutions, Inc.
Coordination of Shipment (Personnel/Organization): FOL (TBD)/TtEC or Plant Operator (Fred Mattison)/ECOR Solutions, Inc.
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodian, Test America – Pittsburgh or Air Toxics, Ltd.
Sample Custody and Storage (Personnel/Organization): Sample Custodian, Test America – Pittsburgh or Air Toxics, Ltd.
Sample Preparation (Personnel/Organization): Preparation Laboratory Staff, Test America – Pittsburgh or Air Toxics, Ltd.
Sample Determinative Analysis (Personnel/Organization): Laboratory Technicians, Test America – Pittsburgh or Air Toxics, Ltd.
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): ≤ 1 day prior to shipment to laboratory
Sample Extract/Digestate Storage (No. of days from extraction/digestion) : 60 days from submittal of final data report
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodian, Test America – Pittsburgh or Air Toxics, Ltd.
Number of Days from Analysis: 60 days from submittal of final data report

SAP Worksheet #27 – Sample Custody Requirements Table
(UFP-QAPP Manual Section 3.3.3)

27.1 SAMPLE DESIGNATION AND TRACKING SYSTEM

Each sample collected will be assigned a unique sample tracking number designated by an alphanumeric code that will identify the site and contain a sequential sample number. The sample tracking number will consist of alpha-numeric characters identifying the site, sample medium, location, and date. Any other pertinent information regarding sample identification will be recorded on the sample log sheets or in the field logbooks. The alpha-numeric (A-N) coding to be used in the sample system is described below.

AAAAA	-	AAA	-	AA	-	AAAA or AANN	-	MMDDYYYY
(Site ID)	-	(Area)	-	(Medium)	-	(Location)	-	(Date)

Site identifier:	NWIRP for Naval Weapons Industrial Reserve Plant
Area identifier	GM-38
Medium identifier:	GW for groundwater well samples, PS for process stream water samples, and AIR for air samples from the vapor phase treatment system
Location identifier:	Monitoring well number or location within process stream or vapor phase treatment system
Date:	All samples will be dated to identify the associated sampling period

Example: The water sample collected from the Naval Weapons Industrial Reserve Plant, GM-38 Area from the stream process influent from RW-1 on December 12, 2009 would be identified as: NWIRP-GM-038-PS-IRW1-12122009.

QC Samples collected during a sampling program typically use the same coding system as the environmental samples. Duplicate samples will have "DUP" added to the end of the sample IDs. Additional volumes for laboratory QC samples (MS/MSD samples) have no separate sample identifier codes, but will be designated on the chain-of-custody record and field sheets.

27.2 SAMPLE COLLECTION DOCUMENTATION

A project-specific field logbook will be used to keep daily records of significant events, observations, and measurements during groundwater sampling and process sampling. The field logbook also will be used to document all sampling activities. Logbook entries will be made with indelible ink to provide a permanent record, and any errors in the logbook will be verified, crossed through, and initialed by the person discovering the error. The field logbooks are intended to provide sufficient data and observations to reconstruct events that occurred during sampling activities. Field logbooks should be permanently bound and pre-paginated; designated forms should be used whenever possible to ensure that field records are complete. The following items are examples of information that may be included in a field logbook:

- Name, date, and time of entry
- Names and responsibilities of field crew members
- Name and titles of any site visitors
- Descriptions of field procedures, and problems encountered
- Samples collected at each location
- Sample identification numbers of all samples collected
- Date and time of collection
- Sample collector
- Sample collection method
- Decontamination procedures
- Weather conditions
- Site observations

- Site sketches
- Health and Safety issues including personal protective equipment
- Log of photographs

The following sections outline the information that will be documented in the field according to the medium to be sampled and the activities to be performed. Examples of these forms can be found in Appendix B.

27.3 FIELD SAMPLE HANDLING AND CHAIN-OF CUSTODY PROCEDURES

Custody of samples must be maintained and documented at all times to ensure the integrity of each sample from collection through analysis. An accurate written record is necessary to trace the possession and handling of the sample; this documentation is referred to as the "chain of custody" form. Chain of custody begins when samples are collected in the field and is maintained by storing the samples in secure areas until custody can be passed on. All samples will be delivered to the laboratory accompanied by a chain-of-custody form that will describe the sample identifiers, dates and times of sample collection, analytical parameters, and persons responsible for the sample integrity.

Prior to sample collection, sample containers will be labeled with the sample location number, sampler's name, date, and analytical fraction. Following collection, samples will be placed on ice in a secure cooler or in a box, as applicable, and attended by TtEC personnel or placed in locked vehicles or designated storage areas until shipment to an off-site laboratory.

For aqueous and spent air stripper media samples, the samples will be shipped to the laboratory in coolers packed with bubble wrap, or equivalent packing material, to cushion the samples and prevent breakage. Ice will be added to the coolers to maintain the required temperature (4º C) of the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with a signed custody seal to ensure that chain of custody is maintained. For air samples, the samples will be shipped to the laboratory in boxes with the canister regulators wrapped with bubble wrap, or equivalent packing material, to cushion the regulators and prevent breakage. The boxes will be taped and sealed with a signed custody seal to ensure that chain of custody is maintained. Samples will be shipped to the laboratory via Federal Express to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding times for each analysis are presented in Worksheet #19. This worksheet also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the sample.

Each sample collected will be assigned a unique sampling tracking number, as described above. The sample number, sample collection date and time, and a list of the sample analyses to be performed will be recorded on each container and also on the chain-of-custody form. The chain-of-custody form is a two-part form: the original accompanies the samples to the analytical laboratory, and the copy will be archived in the project files. The following information will be recorded on the chain-of-custody form:

- Project name and number
- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- QC sample designation
- Analysis method
- Special handling instructions
- Destination of samples
- Name, date, time, and signature of each individual releasing the shipping container

The field crew will attempt to identify any potentially high concentration samples on the chain-of-custody form.

27.4 LABORATORY CUSTODY PROCEDURES

The condition of the shipping cooler/box, custody seals, coolant, integrity and condition of the samples and presence and accuracy of the chain-of-custody documentation will be recorded upon sample receipt. The Laboratory Sample Custodian will then log the samples into a computerized Laboratory Information Management System (LIMS). Any discrepancies will be immediately brought to the attention of the Subcontractor Laboratory's Project Manager for discussion and resolution with the TtEC. Login information will be distributed electronically to each laboratory section, where Supervisors track analyses, holding times and due dates. Special technical instructions including customized analyte lists, special reporting limits, and any other pertinent information will also be contained within the LIMS and accessible to all staff.

The process of logging samples into LIMS assigns each sample a unique laboratory identification number in ascending sequence. After assignment of sample numbers and required tests for all samples in a submittal, the LIMS generates login paperwork summarizing each project. The Subcontractor Laboratory's Project Manager will review the sample receipt and login paperwork to assure that the login process was performed correctly.

The LIMS sheet tracks the status of each test and work order according to the project due date and lists the number of incomplete samples in each laboratory section's backlog. As work is completed and reviewed for each test, the updated information will be recorded in the LIMS. This ensures visibility for project status and due dates, and provides a means of tracking samples through the entire analytical process. This also allows project managers to determine the laboratory's capacity by viewing the backlog of samples in house.

All transfers of samples and sample extracts/digestates will be recorded in custody logs. The entire laboratory facility is maintained as a secure, limited access facility. All documents received with a sample delivery group (SDG) and/or generated in the course of the analyses of samples will be kept confidential. Documentation procedures for record content, format, corrections, dates and signatures will be implemented to meet the requirements for legally defensible data.

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Aqueous
Analytical Group	VOCs (Select and TCL)
Analytical Method/ SOP Reference	EPA 624

QC Sample	Frequency/ Number	Method / SOP QC Acceptance Limits		Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Method Blank	1 per batch or 1 per 20 samples	No target analytes > 1/2 the Quantitation Limit		First re-analyze, possibly re-extract batch	Subcontractor Laboratory GC/MS Technician	Bias / Contamination	No target analytes > 1/2 the QL	
Laboratory Control Sample	1 per batch or 1 per 20 samples	Laboratory Defined		Flag Outliers	Subcontractor Laboratory GC/MS Technician	Accuracy	Laboratory Defined	
Matrix Spike/ Matrix Spike Duplicate	1 per batch or 1 per 20 samples	Laboratory Defined		Flag Outliers	Subcontractor Laboratory GC/MS Technician	Accuracy / Precision	Laboratory Defined	
Surrogates	Each sample	4-Bromofluoro-benzene	74-121 %R	Check instrument performance, re-analyze and qualify data	Subcontractor Laboratory GC/MS Technician / Data Reviewer	Accuracy / Bias	4-Bromofluoro-benzene	74-121 %R
		Dibromofluoro-methane	80-120 %R				Dibromofluoro-methane	80-120 %R
		Toluene-d8	81-117 %R				Toluene-d8	81-117 %R
		Dichloroethene-d4	80-120 %R				Dichloroethene -d4	80-120 %R
Internal Standards	Each sample	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration		Check instrument performance, reanalyze and qualify data	Subcontractor Laboratory GC/MS Technician / Data Reviewer	Precision / Accuracy / Bias	Area counts –50% to +100% of Initial Calibration IS or Continuing Calibration IS area counts; Retention times +/- 30 secs of Continuing Calibration	

Matrix	Aqueous
Analytical Group	Mercury
Analytical Method/ SOP Reference	EPA 245.1

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank (Laboratory Reagent Blank)	1 per batch or 1 per 20 samples	No target analytes > 1/2 the Quantitation Limit	Redigest and reanalyze	Subcontractor Laboratory Technician	Bias / Contamination	No target analytes > 1/2 the QL
Laboratory Fortified Blank	1 per batch or 1 per 20 samples	%Recovery 85% - 115%	Redigest and reanalyze	Subcontractor Laboratory Technician	Accuracy / Bias / Contamination	%Recovery 85% - 115%
Duplicate Sample	1 per 20 samples	RPD <=20%	Qualify data	Data Reviewer	Precision	RPD <=20%
Laboratory Fortified Matrix (Spike)	1 per 20 samples	%Recovery 70% - 130%	Perform post-digestion spike analysis, qualify data	Subcontractor Laboratory Technician / Data Reviewer	Accuracy / Bias	%Recovery 70% - 130%
Post-digestion Spike	For compounds outside of QC limits in Matrix Spike	%Recovery 75% - 125%	Qualify data	Data Reviewer	Accuracy / Bias	%Recovery 75% - 125%

Matrix	Aqueous
Analytical Group	TSS
Analytical Method / SOP Reference	SM 2540D

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per ≤ 20 samples	no constituent > QL	Suspend analysis until source rectified	Laboratory Wet Chemistry Technician	Accuracy	no constituent > QL
Laboratory Duplicate Sample	1 per ≤ 20 samples	± 25% RPD	Reanalyze	Laboratory Wet Chemistry Technician	Precision	± 25% RPD

Matrix	Air
Analytical Group	Select VOCs
Analytical Method / SOP Reference	TO-15

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per 24 hours (after CCV/LCS)	No target analytes > Quantitation Limit	Reanalyze	Subcontractor Laboratory Technician	Bias / Contamination	No target analytes > QL
Laboratory Replicate Sample	10% of samples	± 25%RPD (for detections > 5x the QL)	Reanalyze sample a third time. If still exceeds, investigate cause. Qualify data	Subcontractor Laboratory Technician / Data Reviewer	Precision	± 25%RPD
Laboratory Control Sample	After each initial calibration curve and daily, prior to analysis	70 – 130%R (for 90 percent of standard compounds); 60 – 140%R (for 80 percent of non-standard compounds); No recovery may be < 50%%	Check the system, reanalyze standard. Re-prepare standard if necessary. Re-calibrate instrument if criteria cannot be met.	Subcontractor Laboratory Technician	Accuracy	70 – 130%R (for 90 percent of standard compounds); 60 – 140%R (for 80 percent of non-standard compounds); No recovery may be < 50%%
Surrogate Compounds	Each sample	70 – 130%R	Check instrument performance, reanalyze and qualify data	Subcontractor Laboratory Technician / Data Reviewer	Accuracy	70 – 130%R
Internal Standards	Each sample	Area counts ± 40% of Continuing Calibration IS area counts; Retention times ± 33 secs of Continuing Calibration	Check instrument performance, reanalyze and qualify data	Subcontractor Laboratory Technician / Data Reviewer	Precision / Accuracy / Bias	Area counts ± 40% of Continuing Calibration IS area counts; Retention times ± 33 secs of Continuing Calibration

SAP Worksheet #29 -- Project Documents and Records Table
(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Sample Collection Documents and Records • Field logbook (and sampling notes) • Field sample forms (e.g. groundwater purge sheets, operation checklists, etc.) • Chain-of-custody records • Sample shipment airbills • Photographs • Field Task Modification Forms • Sampling and Analysis Plan • Field Sampling SOPs	TtEC project file.
Laboratory Documents and Records • Sample receipt/login form • Sample storage records • Sample preparation logs • Standard traceability logs • Equipment Calibration logs • Sample analysis run logs • Equipment maintenance, testing, and inspection logs • Corrective action forms • Reported field sample results • Reported results for standards, quality control checks, and quality control samples • Data completeness checklists • Sample storage and disposal records • Telephone logs • Extraction/clean-up records • Raw data	TtEC project file, Long-term data package storage at third-party professional document storage firm (Business Records Management, Inc.).
Data Assessment Documents and Records • Field Sampling Audit Checklist (if an audit is conducted) • Analytical Audit Checklist (if an audit is conducted) • Data Review Memoranda	TtEC project file.

Procedures for data handling, management, tracking, and control are described below.

29.1 DATA HANDLING, MANAGEMENT, TRACKING, AND CONTROL

29.1.1 Data Handling and Management - After the groundwater sampling and process sampling events are completed, the groundwater purge sheets and operation checklists will be organized by date and filed in the project files. The field logbooks for this project will be used only for this site and will also be categorized and maintained in the project files after the completion of the sampling. Project personnel completing concurrent field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity.

29.1.2 Data Tracking and Control - The TtEC PM (or designee) is responsible for the overall tracking and control of data generated for the project.

- **Data Tracking**. Data are tracked from its generation to its archiving in the TtEC project-specific files. The Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contract laboratory. Upon receipt of the data packages from the analytical laboratory, the Project

Chemist will oversee the data review effort, which includes verifying that the data packages are complete and results for all samples have been delivered by the analytical laboratory.

- **Data Storage, Archiving, and Retrieval**. The data packages received from the subcontractor laboratory are tracked in the data review logbook. After the data are reviewed, the data packages are entered into the TtEC file system and archived in secure files. The field records including field logbooks, sample log sheets, and chain-of-custody records will be submitted by the FOL to be entered into the file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be returned to the Navy by TtEC.

- **Data Security**. The TtEC project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

SAP Worksheet #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization	Backup Laboratory / Organization
Aqueous	VOCs (Select and TCL)	See Worksheet #18	EPA 624	15 days	Test America – Pittsburgh 301 Alpha Drive Pittsburgh, PA 15238 (412) 963-2435	Not Applicable
Aqueous	Mercury	See Worksheet #18	EPA 245.1	15 days	Test America – Pittsburgh 301 Alpha Drive Pittsburgh, PA 15238 (412) 963-2435	Not Applicable
Aqueous	TSS	See Worksheet #18	SM 2540D	15 days	Test America – Pittsburgh 301 Alpha Drive Pittsburgh, PA 15238 (412) 963-2435	Not Applicable
Air	Select VOCs	See Worksheet #18	TO-15	15 days	Air Toxics, Ltd. 180 Blue Ravine Road, Suite B Folsom, CA 95630 (800) 985-5955	Not Applicable
Spent Air Stripper Packing/ Filter /Adsorber Media	TCLP VOCs	See Worksheet #18	SW-846 1311/8260B	15 days	Test America – Pittsburgh 301 Alpha Drive Pittsburgh, PA 15238 (412) 963-2435	Not Applicable
Spent Air Stripper Packing/ Filter /Adsorber	TCLP Metals	See Worksheet #18	SW-846 1311/6010C/ 7470A	15 days	Test America – Pittsburgh 301 Alpha Drive Pittsburgh, PA 15238 (412) 963-2435	Not Applicable

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization	Backup Laboratory / Organization
Media						

SAP Worksheet #31 -- Planned Project Assessments Table
(UFP-QAPP Manual Section 4.1.1)
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Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)	Person(s) Responsible for Monitoring Effectiveness of CA
Health and Safety	1 per contract year	Internal	TtEC	Determined at the time of the audit. Will be a TtEC corporate H&S representative or qualified designee	TtEC PM (Stavros Patselas)	Auditor and Health and Safety Manager	Health and Safety Manager (Grey Coppi)
Laboratory Systems Audit	Every 18 months	External	DoD ELAP	Determined at the time of the audit. Will be a Navy representative or qualified designee	Laboratory QA Manager	Laboratory QA Manager	Laboratory QA Manager
Field Sampling Systems Audit	1 per contract year *	Internal	TtEC	Determined at the time of the audit. Will be a TtEC corporate QA representative or qualified designee	TtEC PM (Stavros Patselas)	Auditor and QAM	QAM
Field Supervision	Daily during sampling events	Internal	TtEC	TtEC FOL (TBD)	TtEC FOL (TBD)	TtEC FOL and Field Crew	TtEC FOL, PM, QAM
Project Supervision	Daily	Internal	TtEC	TtEC PM (Stavros Patselas)	TtEC FOL (TBD)	TtEC PM and TtEC FOL	TtEC PM and TtEC FOL

*Whether an audit is conducted or not is determined at the program level, not the project level. The remainder of the table explains how an audit will be handled if an audit occurs.

System audits will be performed as appropriate to ensure that the work is being implemented in accordance with the approved project SOPs and in an overall satisfactory manner.

- During the groundwater sampling, the TtEC FOL will supervise the field operations. The FOL will perform a daily check to ensure the equipment is properly decontaminated, samples are collected and handled properly, and fieldwork is accurately and neatly documented. Corrective actions will be implemented immediately if any non-compliance is detected.

- During the plant operations, the Plant Operator will supervise the plant operations. The Plant Operator will perform checks to ensure the equipment is operating properly, samples are collected according to schedule, and changes are accurately and neatly documented. Corrective actions will be implemented immediately if any non-compliance is detected.

- System audits of the laboratory will be performed regularly and in accordance with DoD ELAP guidance and DoD QSM (2009), as provided in the Laboratory Quality Assurance Plan (LQAP).

- The data reviewer(s) will review the data to ensure that the analytical results were obtained through the approved methodology, and the appropriate levels of QC were followed. The data review effort will be supervised by the TtEC Project Chemist or designee.

The TtEC PM will oversee the FOL, Plant Operator, and data reviewers, and check that management of the acquired data proceeds in an organized manner.

An independent performance audit of field activities may be conducted at the discretion of and under the direction of the QAM. If a formal field audit is conducted, the QAM (or designee) will check that sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures, are being performed in accordance with the approved project planning documents and SOPs. These audits and laboratory systems audits will identify the following:

- The assessed entity (e.g., field crew, office personnel, etc. and the associated project, field event, office, etc.)
- Whether the audit is internal or external
- Location and date(s) of assessment
- Assessment team members
- Type of assessment
- Scope of assessment
- Documents to be reviewed
- Notification dates
- Proposed assessment schedule
- Assessment number
- Contract number

Assessment findings that require corrective action initiate a sequence of events that include documentation of deficiencies, notification of findings, request for corrective action, implementation of corrective action, and follow-up assessment of the corrective action effectiveness. The procedures for handling any SAP deviations and project deficiencies that are identified through the planned project assessments are summarized in Worksheet #32.

Potential problems may involve non-conformance with the SOPs and/or analytical procedures established for the project or other unforeseen difficulties. Any person identifying a condition adverse to project quality will notify the PM. The PM, with the assistance of the QAM, will be responsible for developing and

initiating appropriate corrective action. If the identified deficiencies involve field work, this will be done through the FOL; if the deficiencies involve the laboratory, this will be done through the Laboratory PM/QA Manager. The corrective actions will require follow-through to the point of verifying that the corrective action has been effective. Corrective actions may include resampling and/or reanalyzing samples or amending or adjusting project procedures. If warranted by the severity of the problem (e.g., if a change in the approved plan is required), the Navy will be notified in writing and the Navy's approval will be obtained before any change is implemented. Minor changes will be documented for the main file by the TtEC PM. Additional work that depends on a non-conforming activity will not be performed until the problem has been eliminated. The overall corrective action responsibility for system audits will reside with the PM. The overall corrective action responsibility for field audits will reside with the TtEC QAM.

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses
(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Time Frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Time Frame for Response
Field Supervision	Site log book, groundwater purge sheets, plant operation checklists	TtEC PM (Stavros Patselas), FOL (TBD), Plant Operator (Fred Mattison)	Immediately	Entry in site log book	TtEC PM (Stavros Patselas), TtEC FOL (TBD)	24 hours
Project Supervision	Written report	TtEC Program Manager (Andy Bolt)	Monthly	Written memorandum	TtEC Program Manager (Andy Bolt)	Within a week of notification
Field Sampling System Audit	Audit checklist and written audit finding summary	TtEC PM (Stavros Patselas), TtEC FOL (TBD), Plant Operator (Fred Mattison), and TtEC Program Management (Andy Bolt)	Dependant on the finding, if major a stop work may be issue immediately, however if minor within 1 week of audit	Written memorandum	TtEC QAM (George Sze), Auditor, and Program Manager (Andy Bolt)	Within 48 hours of notification
Laboratory System Audit	Written audit report	Laboratory Manager and QA Manager	Not specified by DoD ELAP	Letter	DoD ELAP	Specified by DoD ELAP

SAP Worksheet #33 -- QA Management Reports Table
(UFP QAPP Manual Section 4.2)

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
Data review report	Per SDG	Within 3 weeks of receipt of laboratory data	TtEC Data Reviewers	TtEC PM (Stavros Patselas) and TtEC project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately upon detection of problem	TtEC QAM (Jonathan Dziekan)	TtEC PM, QAM, and project file
Discharge Monitoring Reports	Monthly for duration of the GWTP operations	Monthly	TtEC PM (Stavros Patselas)	NAVFAC RPM (Lora Fly), NYSDEC Project Engineer (Steven Scharf), and TtEC project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem	Subcontracted Laboratory QA Manager	TtEC PM, QAM, and project file

SAP Worksheet #34 -- Verification (Step I) Process Table

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification
Chain-of-custody forms	The TtEC FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and that the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, Project Manager, and data reviewers.	Internal	TtEC sampler and FOL
SAP sample tables	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	TtEC FOL or designee
Chain-of-custody forms	The laboratory sample custodian will review the sample shipment for completeness and integrity, and will sign accepting the shipment. The TtEC Project Chemist, data reviewers, and/or designee will check that the chain-of-custody form was signed/dated by the TtEC FOL or designee relinquishing the samples and also by the laboratory sample custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory sample custodian 2 - TtEC Project Chemist, data reviewers, and/or designee
Analytical data package	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QA Manager will sign the case narrative for each data package.	Internal	Laboratory QAM
Analytical data package	The data package will be verified for completeness by TtEC data reviewers. Missing information will be requested from the laboratory, and the data review will be suspended until missing data are received.	External	TtEC Project Chemist, data reviewers, and/or designee
Electronic data deliverables	The electronic data will be verified against the chain-of-custody form and hard copy data package for accuracy and completeness.	External	TtEC Project Chemist, data reviewers, and/or designee

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table
(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs	Ensure that all sampling SOPs were followed and any field deviations were documented.	TtEC PM, Plant Operator, FOL, or designee
IIa	Analytical SOPs	Ensure that the laboratory followed the analytical SOPs cited in the SAP and any method deviations were approved by TtEC and documented in the case narrative.	TtEC Project Chemist, data reviewers, and/or designee
IIa	Chain-of-custody	Ensure that the custody and integrity of the samples were maintained from collection to analysis and the custody records are complete and any deviations are recorded.	TtEC Project Chemist, data reviewers, and/or designee
IIa	Holding times	Ensure that the samples were shipped and stored at the required temperature and that sample pH for chemically preserved samples meet the requirements listed in Worksheet #19. Verify that the analyses were performed within the holding times listed in Worksheet #19.	TtEC Project Chemist, data reviewers, and/or designee
IIa	Data results	Check the summary form results against the raw data. Check calculations for accuracy.	TtEC Project Chemist, data reviewers, and/or designee
IIa	Standards	Ensure that the standards used in the field and laboratory are traceable and meet the contract, method, and procedural requirements.	TtEC Project Chemist, data reviewers, and/or designee
IIa/IIb	Laboratory data results for accuracy	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the measurement performance criteria (MPC) listed in Worksheets #12 and #28 were met for all field samples and QC analyses. Verify that field QC samples were collected (if applicable) and analyzed and that the analytical QC criteria set up for this project were met.	TtEC Project Chemist, data reviewers, and/or designee
IIa/IIb	Laboratory duplicate analyses for precision	Ensure laboratory precision by checking the RPD or percent difference values from laboratory duplicate analyses, matrix spike/matrix spike duplicates, and laboratory control sample/laboratory control sample duplicates. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheets #12 and/or #28.	TtEC Project Chemist, data reviewers, and/or designee
IIa/IIb	Sample results for representativeness	Validate that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	TtEC Project Chemist, data reviewers, and/or designee
IIb	Project Quantitation Limits for sensitivity	Validate that the project QLs and MDLs listed in Worksheet #15 were achieved.	TtEC Project Chemist, data reviewers, and/or designee
IIa/IIb	Project action limits	Discuss the impact of matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as no-detected. Document this usability issue and inform the Project Manager.	TtEC Project Chemist, data reviewers, and/or designee

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIb	Analytical data deviations	Determine the impact of any deviation from sampling or analytical methods and SOP requirements and matrix interference effects on the analytical results.	TtEC Project Chemist, data reviewers, and/or designee
IIb	Sample coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations.	TtEC FOL, Plant Operator, or designee
IIa/IIb	Data review report	Review of: • chain-of-custody and sample receipt documents to verify sample identities. • sample log-in documents to verify any potential problems with sample custody, integrity, preservation, labeling, etc. • field blank data to ascertain any problems with container or preservative contamination, or field contamination. • method blank data to determine the presence and approximate concentration of sources of contamination in the analytical process. • matrix spike data as a measure of matrix effects and analytical precision. • field and laboratory duplicate data as a measure of sampling technique applicability, homogeneity, and analytical precision. • standard reference material or laboratory control sample data as a measure of analytical accuracy. Data will be compared to the certified acceptable ranges of analytical values. • sample dates, extraction/digestion dates, and analysis dates to determine whether maximum holding times were met or exceeded. Where appropriate, data qualifiers will be incorporated into certain data summary tables generated for this project. A brief summary of the data QA/QC review will be included in the final report.	TtEC Project Chemist, data reviewers, and/or designee

1 IIa=Compliance with methods, procedures, and contracts (see Table 10, page 117, UFP-QAPP manual, V.1, March 2005).
IIb=Comparison with measurement performance criteria in the SAP (see Table 11, page 118, UFP-QAPP manual, V.1, March 2005).

SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table
(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator
IIa/IIb	Aqueous	VOCs (Select and TCL)	Criteria for EPA 624 listed in Worksheets #12 and #28. If not included in Worksheet #12 or #28, default to USEPA National Functional Guidelines SOPs/USEPA Region II SOPs.	TtEC Project Chemist (Lynn Arabia), data reviewers, or designee
IIa/IIb	Aqueous	Mercury	Criteria for EPA 245.1 listed in Worksheets #12 and #28. If not included in Worksheet #12 or #28, default to USEPA National Functional Guidelines SOPs/USEPA Region II SOPs.	TtEC Project Chemist (Lynn Arabia), data reviewers, or designee
IIa/IIb	Air	Select VOCs	Criteria for EPA 624 listed in Worksheets #12 and #28. If not included in Worksheet #12 or #28, default to USEPA National Functional Guidelines SOPs/USEPA Region II SOPs.	TtEC Project Chemist (Lynn Arabia), data reviewers, or designee

SAP Worksheet #37 -- Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

Data Usability

37.1 ACCURACY ASSESSMENT
Sample collection accuracy cannot be evaluated because there is no standard by which to judge such accuracy. Instead of a quantitative evaluation of sample collection accuracy, compliance with field SOPs will be the metric.

The accuracy of chemical analyses will be assessed through the use of surrogate spikes, MSs, laboratory control samples (LCSs), calibration check standards, internal standards, and blanks. Blanks will be used to infer the potential for positive biases because of contamination. To assure the accuracy of the analytical procedures, at least 1 of every 20 environmental samples will be spiked with known amounts of target analytes (i.e., MSs) prior to preparation for analysis. The spiked samples will be analyzed and the concentrations of each target analyte observed in the spiked sample will be compared to the reported value of the analyte in the unspiked sample to determine the percent recovery (%R) of the analyte. Control charts are plotted by the laboratory for each target analyte and are kept on matrix- and analyte-specific bases. The %R for a spiked sample is calculated using the following formula:

$$\%R = \frac{\text{Amount in Spiked Sample} - \text{Amount in Sample}}{\text{Known Amount Added}} \text{ X } 100\,\%$$

LCSs and surrogate spikes are also analyzed to assess accuracy. The %R calculation for LCSs and surrogate spikes is as follows:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \text{ X } 100\,\%$$

37.2 PRECISION ASSESSMENT
Laboratory duplicate samples (for inorganic analyses) and MSD samples (for organic analyses) will be prepared and analyzed at a minimum frequency of 1 per every 20 environmental samples per matrix. The RPD between a sample or MS (Sample 1) and its duplicate or MSD (Sample 2) is calculated for chemical analyses using the following formula:

$$RPD = \frac{\left|\text{Amount in Sample 1} - \text{Amount in Sample 2}\right|}{0.5\,(\text{Amount in Sample 1} + \text{Amount in Sample 2})} \text{ X } 100\,\%$$

37.3 COMPLETENESS ASSESSMENT
Completeness for this project will be determined based on the number of sample results for each target analyte and each sample type that are usable as determined through data assessment review. Data values rejected during validation (indicated by an “R” or “UR” flag) will be considered unusable unless additional review and documentation by one or more technical team members demonstrates that the rejection was erroneous. To monitor completeness, the number of usable, valid results for each media and analyte will be counted and compared to the completeness objectives.

Percent completeness will be calculated using the following equation:

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

A completeness of at least 95 percent will be expected to be obtained. If this does not occur, the project team will evaluate the effect of not meeting this completeness goal based on conditions that exist at that time.

37.4 OVERALL USABILITY

Immediately after generation, field data, such as data recorded on groundwater purge sheets or operation checklists, will be examined for errors by the data generator. This examination will compare results to field conditions with an attempt to reconcile any apparent anomalies. The FOL or Plant Operator will be responsible for reviewing the field logs each day of field work to verify that the data appear reasonable based on field conditions. The FOL or Plant Operator will look for gross inconsistencies among field data sheets.

Laboratory data will be examined upon receipt from the laboratory in a series of evaluations. The project team will be responsible for evaluating data usability. Depending on project constraints after data collection, this may be done by a subgroup of team members issuing a data usability report to the rest of the team for concurrence prior to completing the monthly Discharge Monitoring Report or it could require inclusion of a data usability assessment section in the report that is submitted to the team for concurrence. This decision will be made by the NAVFAC RPM in conjunction with the team.

After data review, the data will be reconciled with data quality objectives (DQOs) to determine whether sufficient data of acceptable quality are available for decision making. A series of inspections and analyses will be performed to estimate several of the data set characteristics. The evaluations will include simple summary statistics for target analytes, such as maximum concentrations, minimum concentrations, numbers of samples exhibiting no detectable analytes, number of samples exhibiting detectable analytes, and proportion of samples with detectable and undetectable analytes. The data will be presented in a tabular format. These inspections and analyses will be designed to:

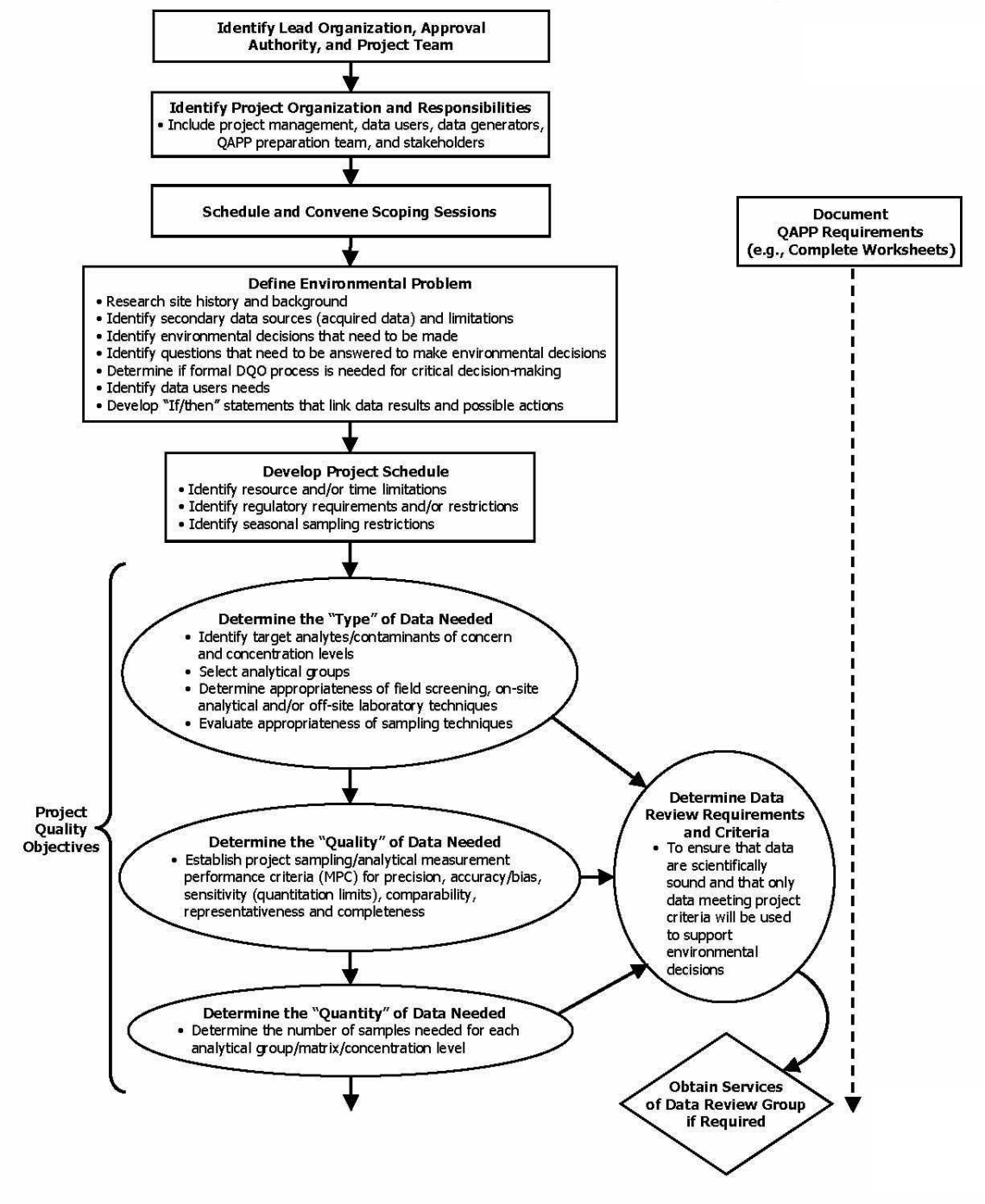
- Identify deviations, if any, from the field sampling SOPs.
- Identify deviations, if any, from the laboratory analytical SOPs.
- Identify deviations, if any, from the SAP.
- Identify deviations, if any, from the data review process.
- Identify and explain the impacts of elevated MDLs, instrument detection limits, and QLs, especially if MDLs exceed project action limits (PALs).
- Identify unusable data (i.e., data qualified as “R”).
- Evaluate the effects of “J” qualified data on data usability and decision making.
- Evaluate project assumptions.
- Evaluate adherence to investigation objectives and decision rules.
- Ensure completion of corrective actions.
- Evaluate effects of deviations from planned procedures and processes on the interpretation and utility of the data.
- Identify remaining data gaps.

For data summaries and mathematical manipulations, analytes that are not detected will be represented by a concentration equal to one-half the sample-specific MDL (inorganics) or QL (organics).

If necessary, investigation objectives may be revised in anticipation of additional data collection.

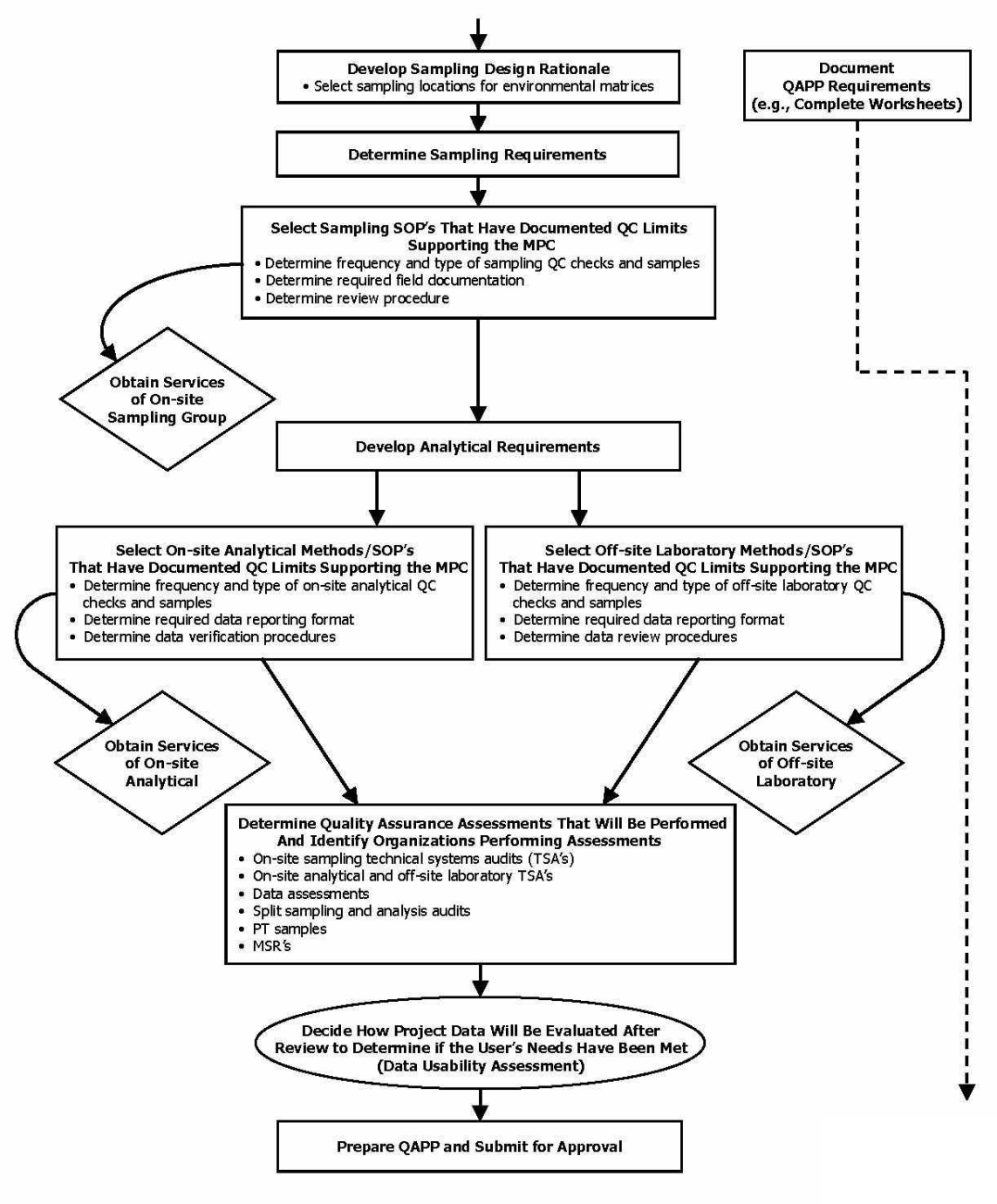
Figures

FIGURE 11-1
SYSTEMATIC PLANNING PROCESS
OPERATION, MAINTENANCE, AND MONITORING PLAN QAPP FOR THE GM-038 GROUNDWATER TREATMENT PLANT



Continued on Page No. 2

FIGURE 11-1
SYSTEMATIC PLANNING PROCESS
OPERATION, MAINTENANCE, AND MONITORING PLAN QAPP FOR THE GM-038 GROUNDWATER TREATMENT PLANT



Appendix A

Field Standard Operating Procedures

Sampling of Process Aqueous Samples (SOP 001)

Process water samples will be collected from various sampling ports within the GWTP building according to the following procedure:

1. Be aware that the nominal process water flow rate through the GWTP is 1100 gallons per minute and that the process water pipelines are under pressure (up to 60 psig). Be aware that the process water contains chlorinated VOCs and follow the proper health and safety guidelines as identified in the SHSP.

2. Partially open the ball valve at the sample port for a few seconds and collect the process water in a 5-gallon bucket in order to flush out any dead zones. Close the ball valve at the sample port and empty the water in the bucket into the GWTP sump.

3. Partially open the ball valve at the sample port for a few seconds once more and collect the process water in a dedicated clean glass beaker. Close the ball valve at the sample port and quickly transfer an appropriate volume of the sample from the beaker into the proper sample vials and bottles.

4. Samples for VOCs must be collected first. The sample vials and bottles should be preserved and filled according to the procedures specified below and in the QAPP.

5. Fill all sample vials and bottles by allowing the water to flow gently down the inside of the vial or bottle with minimal turbulence. Cap each vial or bottle as it is filled.

6. Preserve and label the samples, and record them on the Chain of Custody form and in the field logbook. Place the sample vials and bottles immediately into a cooler for shipment and maintain at 4^{o}C.

7. The filling and preservation procedures will be:

    - VOCs - Determine the amount of 1:1 HCl preservative required to adjust the pH of the sample to less than 2 in an extra 40 ml glass vial. Add this volume to the empty 40 ml vials prior to sampling. Fill each container with sample to just overflowing so that no air bubbles are entrapped inside. If effervescence occurs, submit the sample without preservative and note on the chain of custody form.

    - Other Parameters - Fill each container and preserve immediately as required. To test for pH, pour a minimal portion of sample onto broad range pH paper to verify that the appropriate pH level has been obtained.

Sampling of Process Vapor Samples (SOP 002)

Process vapor samples will be collected from the four sampling ports in the VGAC unit within the GWTP building according to the following procedure:

1. This procedure involves the collection of a 30-minute integrated sample using 6-liter Summa canisters supplied by the laboratory. Be aware that the nominal process vapor flow rate through the GWTP is 8,000 cubic feet per minute and that the process vapor pipelines are under pressure (up to 50 inches of water or 94 mm of Hg). Be aware that the process vapor contains chlorinated VOCs and follow the proper health and safety guidelines as identified in the HASP. Be aware that the Summa canisters and associated hardware are expensive containers that need to be handled with special care.

2. Verify the initial vacuum of the canister as received from the laboratory utilizing the following steps. Confirm that the valve on the canister is closed. Remove the brass cap from the canister and attach the critical orifice flow controller to the canister. Attach the brass cap to the other end of the flow controller. After ensuring that the ¼ inch Swagelok fittings are tight using a 9/16 inch wrench and that you have a closed leak-free train, quickly open and close the canister valve. Read the vacuum on the built-in gauge on the flow controller. The initial vacuum should be greater than 25 in of Hg. If this is not the case, do not use that canister for sampling and call the laboratory to arrange for a replacement. Record the gauge reading in the "initial vacuum" column on the chain of custody form.

3. Connect a purge line to the sample port making sure that the other end is vented outside the GWTP building. Partially open the ball valve at the sample port for a few seconds and allow the line to purge in order to flush out any dead zones. Close the ball valve at the sample port. Disconnect the purge line. UNDER NO CIRCUMSTANCES SHOULD THE VAPORS FROM THE SAMPLE PORT BE VENTED INSIDE THE GWTP. Some of the chlorinated VOCs can be immediately dangerous to life and health.

4. Remove the brass cap from the flow controller and connect the sample train to the sample port. After ensuring that the ¼ inch Swagelok fittings are tight using a 9/16 inch wrench, that you have a closed leak-free train, and that the canister and flow controller are properly supported, quickly open the canister valve (1/2 turn) and the ball valve on the sample port.

5. Monitor the integrated sampling process periodically. After 30 minutes, record the "final vacuum" on the chain of custody form by reading the vacuum on the built-in gauge on the flow controller. Close the canister valve and the ball valve on the sample port.

6. Detach the sampling train from the sample port. Detach the flow controller from the canister and replace the brass cap on the canister. Fill out the canister sample tag and log book making sure that the information matches that recorded on the chain of custody form. DO NOT attach any labels to the surface of the canister or write on the canister.

Sampling of Process Vapor Samples (SOP 002) [cont'd]

7. Return the canisters and the flow controllers to the laboratory in the boxes and packaging provided. Place the chain of custody form (after retaining the appropriate copies) in the box with the canister. Tape the box shut and place custody seals at each opening.

Water Level Measurement (SOP 003)

Static water level measurements will be taken in the 14 installed monitoring wells prior to each groundwater sampling event. Additional rounds of measurements may be collected throughout the field activities under the direction of the FOL.

Water level measurements will be conducted in accordance with the following procedure:

1. Groundwater level measurements will be collected from all monitoring wells primarily using an electronic water level indicator. An interface probe will also be used during the initial measurement round and periodically through the program to check for the presence of free product. Water levels will be measured, relative to surveyed datum (i.e., top of well riser), at a specific mark on the casing, to the nearest 0.01 foot.

2. Electronic water level indicators will preferably be the type with water level markings on the cable at increments of 0.01 foot or less.

3. All electronic water level measurements will be recorded in the appropriate field logbook or data sheet.

4. The electronics of the water level indicator will be checked prior to the commencement of measurements with a jar of water and the depths calibrated on the ground against a steel tape.

5. The water level indicator cable, tape and probe will be decontaminated between wells by rinsing with deionized water (see SOP 006).

Field Parameter Measurements During Groundwater Sampling (SOP 004)

Field parameters (temperature, pH, turbidity, ORP, specific conductance, and dissolved oxygen) will be monitored during purging of the monitoring wells utilizing a Horiba® water quality meter or equivalent. Measurements will be conducted in accordance with the manufacturer's instructions and the following procedure:

1. Calibrate the water quality meter as per manufacturer's instructions.

2. For low flow purging of the monitoring wells:

    - Attach a flow-through cell to the Teflon-lined polyethylene tubing. Position the water quality meter probe in the flow-through cell. Begin purging the monitoring well, following SOP 005 (Groundwater Sampling [Low Flow Purge Procedure]).

    - After the cell has been "flushed" at least twice, begin monitoring the field parameters, and continue approximately every 3 to 5 minutes during purging. All water quality measurements will be recorded in the appropriate field logbook or on a well purge data sheet.

    - When the indicator parameters have stabilized for three consecutive readings (see Step 11 of SOP 005 (Groundwater Sampling [Low Flow Purge Procedure])), the well is considered stabilized and ready for sample collection. Remove the flow-through cell from the tubing.

3. The probe of the water quality meter will be decontaminated between wells by rinsing with deionized water (see SOP 006).

Groundwater Sampling [Low Flow Purge Procedure] (SOP 005)

Groundwater samples will be collected from 9 monitoring wells installed at the Site. Groundwater samples will be obtained starting at the least contaminated well and proceeding systematically to the well likely to be most contaminated.

1. Check and record the condition of the well for any damage or evidence of tampering.

2. Remove the well cap.

3. Measure well headspace with a PID and record the reading in the field logbook.

4. Measure and record the depth to water, as stated in SOP 003 (Water Level Measurement), and record the measurement in the field logbook. Do not measure the depth to the bottom of the well at this time (to avoid disturbing any sediment that may have accumulated). Obtain depth to bottom information from installation information in the field logbook or drilling logs. Calculate volume of the water column.

5. Lay out plastic sheeting and place the monitoring, purging and sampling equipment on the sheeting. To avoid cross-contamination, do not let any downhole equipment touch the ground.

6. Re-check and record the depth to water after approximately 5 minutes at the well location. If the measurement has changed more than 0.01 foot, check and record the measurement again, then begin well purging.

7. Attach and secure the Teflon-lined polyethylene tubing to the low-flow submersible pump. As the pump is slowly lowered into the well, secure the safety drop cable, tubing, and electrical lines to each other using nylon stay-ties placed approximately 5 feet apart.

8. Set the pump at approximately the middle of the screen and/or the best depth based on the stratigraphy of the well. Be careful not to place the pump intake less than 2 feet above the bottom of the well as this may cause mobilization of any sediment present in the bottom of the well. Start pumping the well at 0.2 to 0.5 liters per minute.

9. Monitor the water level in the well periodically during pumping, and ideally the pump rate should equal the well recharge rate with little or no water level drawdown in the well (drawdown shall be 0.3 foot or less). There should be at least 1 foot of water over the pump intake so there is no risk of the pump suction being broken, or entrapment of air in the sample. Record the pumping rate adjustments and depth(s) to water in the logbook. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump (0.1 to 0.2 liters per minute) to avoid purging the well dry. However, if the recharge rate of the well is very low and the well is purged dry, then wait until the well has recharged to a sufficient level and collect the appropriate volume of sample with the submersible pump.

Groundwater Sampling [Low Flow Purge Procedure] (SOP 005) [cont'd]

10. Purge the well at a low-flow rate (from 0.2 to 0.5 liters per minute). During purging, monitor the field parameters (temperature, pH, turbidity, Eh, specific conductance, and dissolved oxygen) approximately every 3 to 5 minutes. A flow-through cell will be used to monitor the field parameters (see SOP 004). Begin measuring field parameters after the flow-through cell has been "flushed" with groundwater twice.

11. The well is considered stabilized and ready for sample collection when the indicator parameters have stabilized for three consecutive readings, as follows:

    - 0.1 for pH
    - 3 percent for specific conductance
    - 10 percent for dissolved oxygen
    - 10 percent for turbidity
    - 10 mV for ORP

    Dissolved oxygen and turbidity usually require the longest time to achieve stabilization. The pump must not be removed from the well between purging and sampling.

12. Once the field parameters have stabilized, collect the samples directly from the end of the tubing. Volatiles and analyses that degrade by aeration must be collected first. The bottles should be preserved and filled according to the procedures specified below and in the QAPP.

13. Fill all sample bottles by allowing the pump discharge to flow gently down the inside of the bottle with minimal turbulence. Cap each bottle as it is filled.

14. The filling and preservation procedures will be:

    - VOCs – The laboratory will determine the approximate amount of 1:1 HCl preservative required to adjust the pH of a sample to less than 2 and will add this volume to the empty 40 ml vials prior to sampling. Fill each container with sample to just overflowing so that no air bubbles are entrapped inside. Test one vial to determine if the pH is below 2. If effervescence occurs, submit the sample in bottleware without preservative and note on the chain of custody form.

    - Other Parameters - Fill each container, careful of any preservative added by the laboratory. To test for pH, pour a minimal portion of sample onto broad range pH paper to verify that the appropriate pH level has been obtained.

15. Label the samples and record them on the chain of custody. Place immediately into a cooler for shipment and maintain at 4EC.

Groundwater Sampling [Low Flow Purge Procedure] (SOP 005) [cont'd]

16. Carefully remove the pump assembly from the well. The Teflon-lined polyethylene tubing will be dedicated to each well. The tubing should be placed in a large plastic garbage bag, sealed, and labeled with the appropriate well identification number.

17. After sampling is complete, measure the total depth of the well.

18. Close and lock the well.

Decontamination – Field Instrumentation - Probes, Water Quality Meters, etc. (SOP 006)

Field instrumentation (such as water level probes, water quality meters, etc.) will be decontaminated between sample locations by rinsing with deionized water. If visible contamination still exists on the equipment after the rinse, an Alconox detergent scrub will be added, and the probe thoroughly rinsed again.

Decontamination of sampling equipment will be kept to a minimum in the field and wherever possible, dedicated disposable sampling equipment will be used. Any decontamination fluids generated will be stored in U.S. Department of Transportation (DOT)-approved 55-gallon drums or in an on-site storage tank (liquids only) until disposal. Personnel directly involved in equipment decontamination will wear appropriate protective clothing, as stated in the HASP.

Decontamination – Non-Disposable Chemical Sampling Equipment (SOP 007)

Wherever possible, disposable equipment will be used for groundwater and process water sampling events and decontamination will not be required. Should non-disposable sampling equipment be used, it will be decontaminated prior to collecting each sample. Decontamination of non-disposable sampling equipment used to collect samples for chemical analyses (i.e., pumps, beakers, etc.) will be conducted as described below:

1. Remove all visible contaminants using laboratory detergent (i.e., Alconox) and potable water scrub.

2. Potable water rinse.

3. De-ionized water rinse.

4. Air dry.

5. Wrap or cover exposed ends of equipment with aluminum foil for transport and handling.

Decontamination of sampling equipment will be kept to a minimum in the field and, whenever possible, dedicated disposable sampling equipment will be used. Decontamination fluids will be stored in appropriately sized DOT-approved containers or in an on-site storage tank (liquids only) until transported off-site for disposal. Personnel directly involved in equipment decontamination will wear appropriate protective clothing, as stated in the HASP.

Sampling of Spent Air Stripper Packing/Filter/Adsorber Material (SOP 008)

Spent air stripper packing/filter/adsorber material will be collected prior to disposal according to the following procedure:

1. To obtain a sample of the spent air stripper packing/filter/adsorber material, use a disposable scoop to dig into the material at random locations.

2. Collect a sufficient volume of material to fill the applicable bottleware and place in a disposable aluminum pan. Homogenize the material thoroughly. Separate the material into the appropriate containers for the analysis of TCLP VOCs and TCLP metals.

3. Label the samples and record them on the chain of custody form and in the field logbook. Place the sample vials and jars immediately into a cooler for shipment and maintain at 4^{o}C.

Appendix B

Field Forms

APPENDIX B-1
SAMPLE LABEL AND TtEC CUSTODY SEAL (TYPICAL)
GM-038 GROUNDWATER TREATMENT PLANT

Sample No.
Sample Location
Matrix / Type
Date / Time
Sampler
Analysis
Preservation

TETRA TECH **SIGNATURE: _____ DATE: _____ TIME: _____**	**SAMPLE CUSTODY SEAL**	**SEAL BROKEN** **BY: _____** **DATE: _____**

APPENDIX B-2
TtEC CHAIN OF CUSTODY FORM (TYPICAL)
GM-038 GROUNDWATER TREATMENT PLANT

TETRA TECH

CHAIN OF CUSTODY RECORD

PROJECT					NO. CONTAINERS										REMARKS OR SAMPLE LOCATION	PRESERVATION	
SAMPLERS: (Signature)																ICED	SPECIFY CHEMICALS ADDED AND FINAL pH IF KNOWN
SAMPLE NUMBER	DATE	TIME	COMP.	GRAB													

Relinquished by: (Signature) (1)	Date / Time	Received by: (Signature)	Relinquished by: (Signature) (4)	Date / Time	Shipped via:
Relinquished by: (Signature) (2)	Date / Time	Received by: (Signature)	Received for Laboratory by: (Signature)	Date / Time	Shipped via:
Relinquished by: (Signature) (3)	Date / Time	Received by: (Signature)	Remarks		

APPENDIX B-3
EQUIPMENT/INSTRUMENT CALIBRATION AND MAINTENANCE FORM (TYPICAL)
GM-038 GROUNDWATER TREATMENT PLANT

PROJECT: _____ **PROJECT No.:** _____ **DATE:** _____ **SHEET** _____ **of** _____

INSTRUMENT (NAME / MODEL NO. / SERIAL NO.): _____

MANUFACTURER: _____ **DATE PURCHASED or LEASED:** _____

CALIBRATION LOGSHEET

CALIBRATION DATE	INITIAL SETTINGS	STANDARD(S) USED	PROCEDURE	ADJUSTMENTS MADE	FINAL SETTINGS	SIGNATURE	COMMENTS

MAINTENANCE LOGSHEET

MAINTENANCE DATE	REASON FOR MAINTENANCE	MAINTENANCE PERFORMED	SIGNATURE	COMMENTS

APPENDIX B-4
FIELD CHANGE REQUEST FORM (TYPICAL)
GM-038 GROUNDWATER TREATMENT PLANT

FCR Number: _____

Field Change Request Title: _____

To: _____ **Location:** _____

Date: _____

Description:

_____

_____

Reason for Change:

_____

_____

Recommended Disposition:

_____

_____

_____	_____	_____
Field Operations Lead (or designee) [print name]	Signature	Date

I have reviewed the above change request, and

[ ] approve the modification.

[ ] do not approve the modification.

The above change request has been discussed with *client* personnel:

[ ] Yes (see below).

[ ] No. The change is minor and does not need *client* concurrence.

_____	_____	_____
Project Manager [print name]	Signature	Date

I have reviewed the above change request, and

[ ] concur with the modification.

[ ] do not concur with the modification.

_____	_____	_____
NAVFAC Remedial Project Manager [print name]	Signature	Date

Distribution:

NAVFAC Remedial Project Manager
TtEC Program Manager
TtEC Project Manager
Field Operations Lead
Project File
Other:

Appendix C

Test America – Pittsburgh Documentation

CERTIFICATE OF ACCREDITATION

ANSI-ASQ National Accreditation Board/ACLASS
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

Test America - Pittsburgh
301 Alpha Drive
Pittsburgh, PA 15238

has been assessed by ACLASS
and meets the requirements of

DoD-ELAP

while demonstrating technical competence in the field(s) of

TESTING

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

ADE-1422
Certificate Number

ACLASS Approval

Certificate Valid: 03/12/2010-03/12/2012
Version No. 001

SCOPE OF DoD-ELAP ACCREDITATION

TestAmerica Pittsburgh

301 Alpha Drive, Pittsburgh PA 15238
Nasreen K. DeRubeis Phone: 412-963-7058

TESTING

Valid to: March 12, 2012 Certificate Number: ADE - 1442

I. Environmental

MATRIX	SPECIFIC TEST or ANALYTE GROUP	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water and Solids	Metals	6010B / C	ICP-AES
Water and Solids	Metals	6020 / 6020A	ICP-MS
Water and Solids	Mercury	7470A and 7471A / B	CVAA
Water and Solids	Hexavalent Chromium with Alkaline Digestion	7196A	Spectrophotometer
Water and Solids	Total Cyanide	9012A / B 9010B / C / 9013	
Water and Solids	Anions	9056A	IC
Water and Solids	Oil and Grease	9071B / 9070A EPA 1664A	Gravimetric
Water and Solids	Organochlorine Pesticides	8081A / B	GC
Water and Solids	Organo-Phosphorus Compounds	8141A / B	GC
Water, Solids, and Oil	PCBs	8082 / 8082A	GC
Water and Solids	Chlorinated Herbicides	8151A	GC

MATRIX	SPECIFIC TEST or ANALYTE GROUP	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water and Solids	Volatiles	8260B	GC/MS
Water and Solids	Semi-Volatiles	8270C / D	GC/MS
Water	Total Organic Carbon	9060/9060A	TOC Analyzer
Water	EDB and DBCP	8011	GC
Water and Solids	PAHs	8310	HPLC
Solids	Total Organic Carbon	Lloyd Kahn	TOC Analyzer
Water and Solids	Sulfide	9030B / 9034	Titration
Water	pH	9040B / C	pH Meter
Solids	pH	9045C / D	pH Meter
Water and Solids	Flashpoint	1020B / AST- D3278-96	Setaflash closed tester
Water and Solids	Flashpoint	1010A / AST- D93-08	Pensky-Martens Closed Flash Tester.
Solids	Percent Moisture	SM 2540G	Balance
Water	Acid Digestion	3005A	FLAA / ICP
Water	Acid Digestion	3010A	FLAA / ICP
Solids	Acid Digestion	3050B	
Solids	Alkaline Digestion	3060A	
Water	Purge-and-Trap	5030B	
Solids	Closed-system Purge-and-Trap	5035	
Solids	Waste Dilution	3585	
Solids	Automated Soxhlet Extraction	3541	

MATRIX	SPECIFIC TEST or ANALYTE GROUP	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Water	Liquid-Liquid Extraction	3510C	
Water	Continuous Liquid-Liquid Extraction	3520C	
Solids	Ultrasonic Extraction	3550C	
Solids	Waste Dilution	3580A	
Water and Solids	Sulfur Cleanup	3660B	
Water and Solids	Gel Permeation Cleanup	3640A	
Water and Solids	TCLP Toxicity Leaching	1311	

Notes:

1. ** = As Applicable*
2. *This scope is part of and must be included with the Certificate of Accreditation No. ADE-1442*

Vice President

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Pittsburgh

SOP No. PT-MS-002, Rev. 15
Effective Date: 08/05/2010

Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS

Methods: SW-846 8260B AND EPA 624

Approvals (Signature/Date):

Kathy Gordon — 08/05/10	Steve Jackson — 08/05/10
Kathy Gordon — Date	Steve Jackson — Date
Technical Specialist	Health & Safety Manager
Nasreen DeRubeis — 08/04/10	Albert F. Vicinie — 08/04/10
Nasreen DeRubeis — Date	Albert F. Vicinie — Date
Quality Assurance Manager	Laboratory Director

1. 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 1, 2, and A-1. For DoD V4.1 refer to SOP PT-QA-029.

1.2. This SOP is applicable to method 8260B and 624. Appendix A present modifications to the procedures in the main SOP that are necessary for analysis of water samples by method 624. For DoD QSM 3.0 requirements refer to SOP PT-QA-025, Implementation of DoD QSM Version 3, January 2006. For DoD QSM 4.1 requirements refer to SOP PT-QA-029, Implementation of DoD QSM Version 4.1, April 2009.

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 250 µg/L for 5 mL standard level waters, 1 to 40 µg/L for low level waters, 5 to 250 µg/kg for low-level soils, and 250 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1, 2, and A-1.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. 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. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be frozen 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 back flushed 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. 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.

6.8.1 The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is received, an LCS/LCSD will be used in the place of an MS/MSD. Refer to the TestAmerica Pittsburgh QC Program document (PT-QA-021) for further details of the batch definition.

3.2. Method Blank

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)

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

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)

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)

CCCs are a representative group of compounds, which are used to evaluate initial calibrations and continuing calibrations. Relative standard deviation (%RSD) for the initial calibration and % drift or % deviation (%D) for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds, which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. Response factors from the initial and continuing calibrations are 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 an antifoaming agent (Dow Corning Antifoam C) can be used. A blank spiked with this agent must be analyzed with the sample to show there is no target interferences induced by this agent. The antifoaming agent is not used routinely. If it needs to be used, approval from Project Manager is obtained, unless prior client approval has been obtained.

5. SAFETY

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

5.2. 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.

5.3. There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.4. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

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
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.			

5.6. 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 become 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 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.

5.8. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.9. 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 or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 uL and larger, 0.006 inch ID needle.

6.2 Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.

6.2. Balance: Top-loading balance capable of weighing 0.01 g

6.3. Glassware:

6.8.1 Vials: 40 mL with screw caps and Teflon liners.

6.8.2 Volumetric flasks: 10 mL, 50 mL and 100 mL, class A with ground-glass stoppers.

6.4. Spatula: Stainless steel.

6.5. Disposable pipettes: Pasteur.

6.6. pH paper: Narrow range.

6.7. Gases: Helium: Ultra high purity, gr. 99.999%.

6.8. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.8.1 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.

6.8.2 Trap: OI # 10

6.8.3 Desorber: The desorber should be capable of rapidly heating the trap to at least 180°C. Many such devices are commercially available.

6.8.4 Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.9 Gas Chromatograph/Mass Spectrometer System:

6.9.1 Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.9.2 Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.9.2.1 Column 1: 20m x 0.18 ID J&W DB-624 or Restek 502.2 with 1 µm film thickness.

6.9.3 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 or 25 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.9.4 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 9.5) Reagent water is obtained from Millipore system. Other methods of preparing reagent water are acceptable.

7.1.3 1:1 HCl

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 is prepared from the stock solution(s) in methanol. The working standard solutions will be prepared monthly with the exceptions of the gases and 2-chloroethylvinyl ether solutions, which will be prepared on a weekly basis. 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 6 for internal standard components.

7.2.3 Surrogate Standards: Refer to Table 7 for surrogate standard components and spiking levels.

7.2.4 Laboratory Control Sample Spiking Solutions: Refer to Table 8 for the normal control LCS components and spiking levels.

7.2.5 Matrix Spiking Solutions: The matrix spike contains the same control components as the LCS. Refer to Table 8.

7.2.6 Tuning Standard: A standard is made up that will deliver up to 50 ng on column upon injection. A recommended concentration of 25 ng/mL of 4-Bromofluoro-benzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Holding time for preserved volatile samples is 14 days from sample collection. Holding times for unpreserved waters is 7 days. Holding time for unpreserved soils requires that they are analyzed or preserved within 48 hours of sampling.

8.2 Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid.

8.3 Several different approaches to sample preservation and storage are presented below. The appropriate procedure selection is subject to project or program specific requirements.

8.4 Solid samples are prepped in a VOA vial with volatile free water and frozen within 48 hours of sampling 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. Terra Core™ kits (from C &G Scientific) can also be used. The kits are shipped to the field. Each kit includes two low level vials, one medium level vial and one bottle for percent moisture. One kit is used per each sample.

8.5 There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore or Terra CoreTM sample. 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 Terra CoreTM kits or field preservation. The water preservation with freezing method is referenced in Method 5035A, Sec 8.2.1.2 and Appendix A table A-1.

8.6 Sample collection for medium level analysis using EnCore or Terra Core™ samplers.

8.6.1 Ship one 5 g EnCore or Terra Core™ sampler per field sample position.

8.6.2 An additional bottle must be shipped for percent moisture determination.

8.7 When the EnCore samples are returned to the lab, extrude the (nominal) 5g sample into a tared VOA vial containing 5 mL methanol. Obtain the weight of the soil added to the vial and note on the label. The surrogate and the matrix spike solution is added at the time of analysis. Terra Core™ samples are already prepared when received at the laboratory.

8.7.1 Prepare an LCS for each batch. Spike the LCS at the time of analysis.

8.7.2 Shake the samples for two minutes to distribute the methanol throughout the soil.

8.7.3 Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4 + 2 °C until analysis.

8.8 Sample collection for medium level analysis using field methanol preservation

8.8.1 A 5 g sample is to be used, add 5 mL methanol to a 40 ml VOA vial. The surrogate and matrix spike solution is added at the time of analysis).

8.8.2 Seal the bottle and attach a label.

8.8.3 Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.8.4 Ship with appropriate sampling instructions.

8.8.5 Each sample will require an additional bottle with no preservative for percent moisture determination.

8.8.6 At client request, the methanol addition and weighing may also be performed in the field.

8.8.7 When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.9 Low level procedure

8.9.1 If low detection limits are required (typically < 50 µg/kg) freezing the EnCore or Terra Core™ may be used. However, it is also necessary to take a sample for the medium level (field methanol preserved or using the EnCore or Terra Core™ sampler) procedure, in case the concentration of analytes in the soil is above the calibration range of the low level procedure.

8.9.2 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).

8.9.3 The soil sample is taken using a 5g EnCore sampling device or Terra CoreTM and returned to the lab. It is recommended that two EnCore or Terra CoreTM 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.

8.9.4 Prepare VOA vials by adding 5 mL of reagent water only.

8.9.5 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.

8.9.6 Weigh the vial to the nearest 0.01g and note the weight on the label.

8.9.7 Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note. Terra CoreTM samples are already prepared when received at the laboratory. Water preserved vials must be frozen.

8.9.8 Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle 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.

8.10 Unpreserved soils

8.10.1 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 SW-846 Method 5030A. It is no longer included in SW-846 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.11 Aqueous samples are stored in glass containers with Teflon lined septa at 4ºC ± 2ºC, with minimum headspace.

8.12 Medium level solid extracts are aliquoted into 4 mL glass vials with Teflon lined caps and stored at 4ºC ± 2ºC. The extracts are stored with minimum headspace.

8.13 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 frozen) is 48 hours.

8.14 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 7 days.

8.15 Regulatory requirements for Acrolein, Acrylonitrile and 2-Chloroethyl vinyl ether

8.15.1 **Acrolein**: Both 40 CFR 136 and SW 846 (chapter 4) have special preservation requirements to adjust pH to between 4-5. For properly preserved samples (pH 4-5) the holding time is 14 days. There are currently no regulatory options for HCL preservation to < 2, however there are options for an unpreserved water sample.

- **40 CFR 136 (Method 624)**

  Unpreserved sample: If Acrolein is a target analyte the holding time is 3 days.
- **SW 846 (Method 8260) Chapter 4**

  SW 846 does not provide guidance on processing of unpreserved samples. However, EPA MICE has interpreted the holding time on an unpreserved sample as 7 days.

8.15.2 Acrylonitrile: Both 40 CFR 136 and SW 846 (chapter 4) have special preservation requirements to adjust pH to between 4-5. For properly preserved samples (pH 4-5) the holding time is 14 days. However, according to 40 CFR 136, the pH adjustment is not necessary for Acrylonitrile therefore the holding time for unpreserved samples is also 14 days.

- **40 CFR 136 (Method 624)**

  Unpreserved sample: If only Acrylonitrile (no acrolein) is a target analyte the holding time is 14 days.

- **SW 846 (Method 8260) Chapter 4**
  SW 846 does not provide guidance on processing of unpreserved samples. However, EPA MICE has interpreted the holding time on an unpreserved sample as 7 days.

8.15.3 2-Chloroethyl-vinyl ether (2-CEVE): According to 40 CFR 136 purgeable halocarbons (2-CEVE's category) do not require acid preservation and the holding time is 14 days. When Aromatics are included as compounds of interest, samples require acid preservation due to rapid breakdown through bio degradation. The method (624) is designed to use unpreserved containers but includes a caveat that refrigeration alone won't suffice for aromatics stored past 7 days. When aromatics are included the method recommends collection of a separate acidified sample aliquot followed by refrigeration up to 14 days. SW846 includes specific information on the handling of this analyte.

8.15.4 Technical Guidance

Acid preservation or pH adjustment

The stability of 2-Chloroethylvinyl ether and Acrolein is reduced when subjected to low pH. It is therefore not recommended that these compounds be analyzed from routinely preserved VOA vials and since there is no reasonable way to achieve a pH between 4 and 5, it is recommended that unpreserved vials be used for analysis of these compounds.

Holding Time

Where Method 624 data are being used for compliance monitoring, the regulatory holding times take precedence (see above discussion and table).

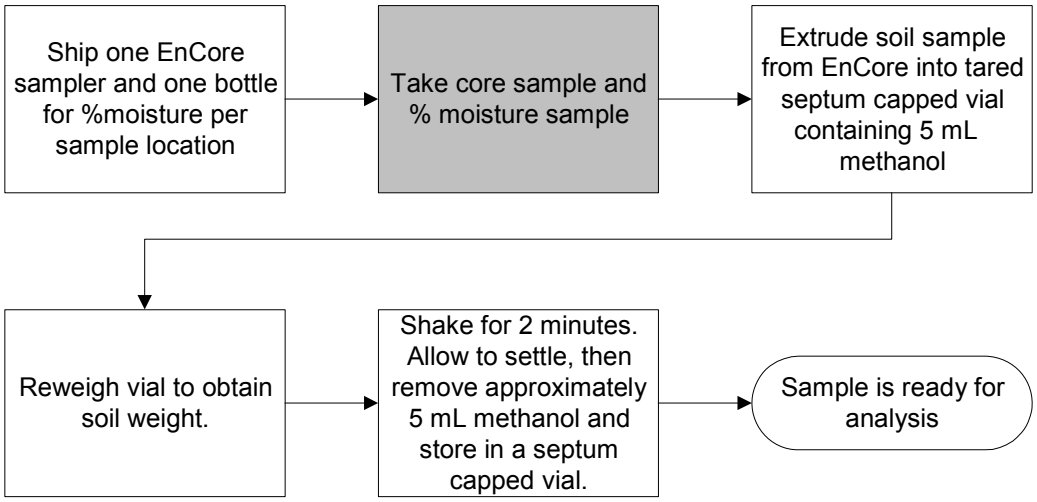
Where Method 624 data are not being generated for compliance purposes, the technical stability of the compounds may be considered. Where the base method stems from SW846, it is allowable to qualify the results. However, the laboratory should make every attempt to analyze samples within the most liberal holding time. To deviate from the regulatory holding times, the following documentation must be maintained:

A. Written confirmation must be obtained from the client that samples are non-compliant.
B. Written approval must be obtained from the client that regulatory holding times may be exceeded (e-mail is acceptable).
C. A method non-conformance statement must be included in the data report.

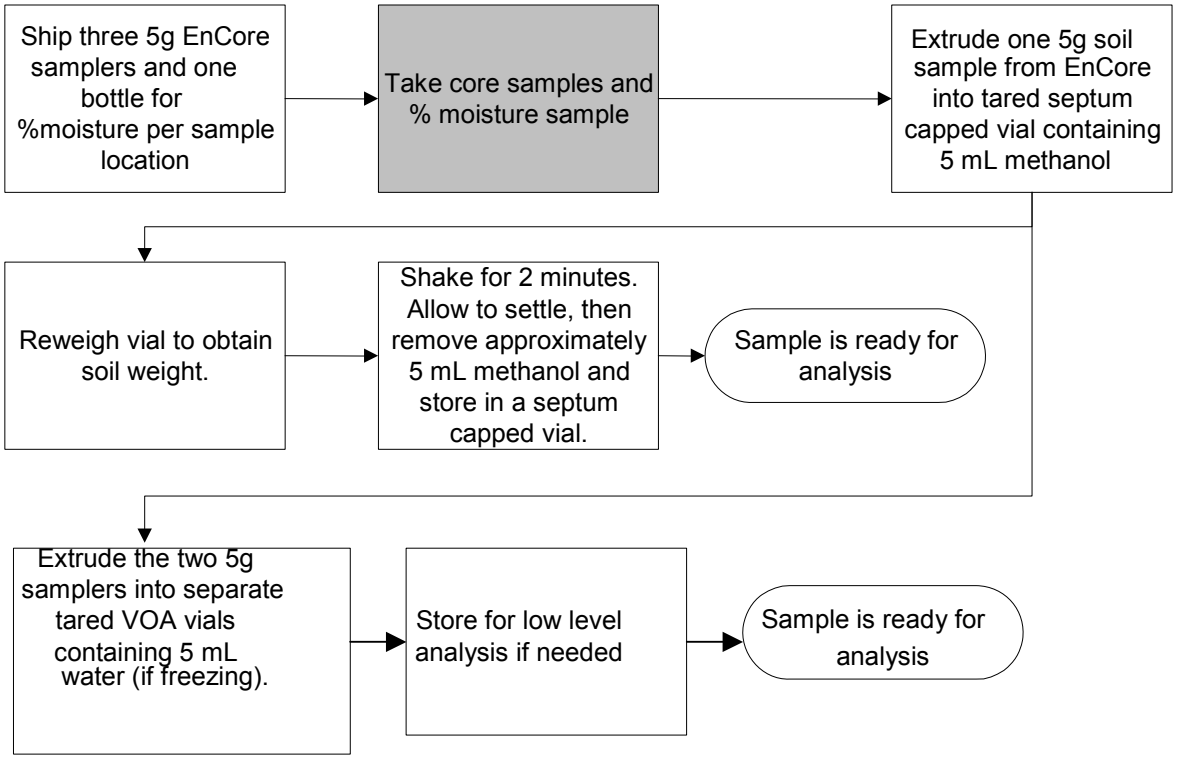
Preservation and Holding Time Table for Volatiles (Dechlorination as needed per Methods)			
Analyte	**Method**	**Preservation**	**Holding Time**
Acrolein	8260	< 6ºC (No HCl)	7 days
	8260	pH 4-5, < 6ºC	14 days
	624	< 6ºC (No HCl)	3 days
	624	pH 4-5, < 6ºC	14 days
Acrylonitrile	8260	< 6ºC (No HCl)	7 days
	8260	pH 4-5, < 6ºC	14 days
	624	< 6ºC (No HCl)	14 days
	624	pH 4-5, < 6ºC	14 days
2-CEVE	8260	< 6ºC (No HCl)	7 days
	624	< 6ºC (No HCl)	14 days

NOTE: If Aromatics are compounds of interest and biological activity is known or suspected to be present, preserved aliquots must be collected.

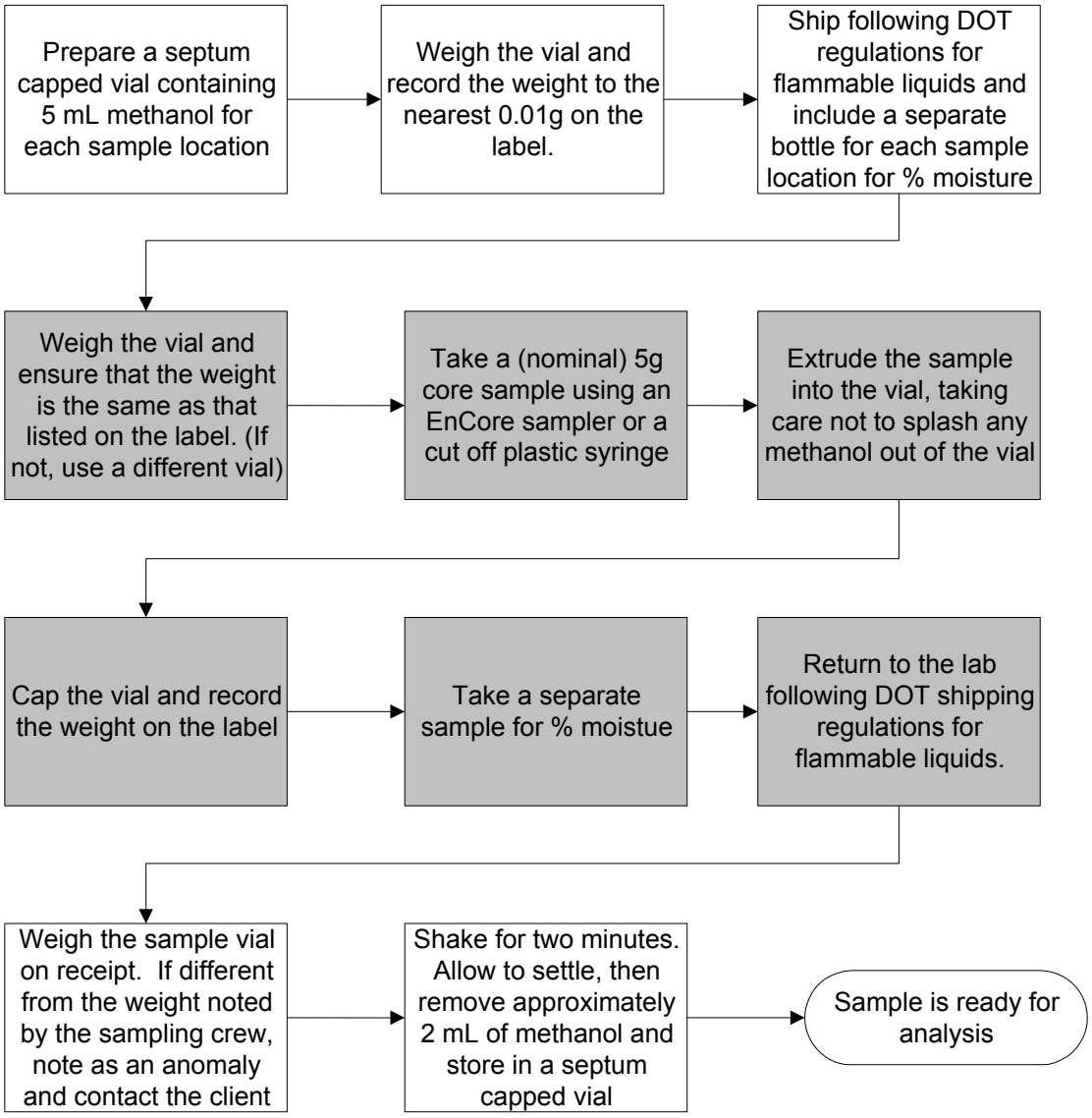
EnCore procedure when low level is not required (field steps in gray)



EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



9 QUALITY CONTROL

9.1 See Document PT-QA-021 "TestAmerica Quality Control Program" for additional detail. For DoD requirements refer to SOP # PT-QA-025, Implementation of DoD QSM Version 3 January 2006, current version and DoD Tables B-1 and B-3. For DoD V4.1 refer to SOP PT-QA-029.

9.2 In-house historical control limits have been determined for surrogates, matrix spikes, and laboratory control samples (LCS). The LCS limits for method 624 are defined in the method and are listed on Table A-2. These limits must be re-checked at least annually. The recovery limits are mean recovery ± 3 standard deviations for surrogates, matrix spikes and LCS. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference ± 3 standard deviations.

9.2.1 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.

9.2.2 Refer to the QC Program document (PT-QA-021) for further details of control limits.

9.3 Surrogates

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)

9.3.1 Check all calculations for error.

9.3.2 Ensure that instrument performance is acceptable.

9.3.3 Recalculate the data and/or reanalyze if either of the above checks reveal a problem

9.3.4 Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem

9.3.5 Samples that have major matrix interference, which is obvious from the chromatogram, will not be rerun for confirmation of matrix interference.

9.3.6 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.

9.3.7 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.

9.3.8 Refer to the TestAmerica Pittsburgh QC Program document (PT-QA-021) for further details of the corrective actions.

9.4 Method Blank

For DoD method blank criteria, see SOP # PT-QA-025 and PT-QA-029. For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. If the first method blank does not meet criteria, a second blank may be analyzed. The method blank must meet criteria before proceeding with sample analyses. For low-level volatiles, the method blank consists of reagent water or 5 grams of Ottawa sand (soil blanks). For medium-level volatiles, the method blank consists of 100 ul of methanol extract into 4.9 mls of reagent water. 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.

9.4.1 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 not more than five times the reporting limit. Such action must be taken in consultation with the client.

9.4.2 Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.4.3 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 done in consultation with the client.

9.4.4 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 sample analysis is free of contamination. All non-conforming blanks will be documented in a non-conformance memo and if reported the reasons for reporting the data will be summarized. For example, if surrogate recoveries are low, re-extraction and/or reanalysis of the blank and affected samples will normally be required. Consultation with the client should take place. If the surrogate recoveries are high and there are target compounds found in the associated sample the samples will require re-extraction and/or reanalysis.

9.4.5 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all compounds detected in the blank are flagged with a "B" in the associated samples, and appropriate comments are made in a narrative to provide further documentation.

9.4.6 Refer to the TestAmerica Pittsburgh QC Program document (PT-QA-021) for further details of the corrective actions.

9.5 Laboratory Control Samples (LCS)

For DoD LCS criteria, see SOP # PT-QA-025 and PT-QA-029. For each batch of samples, analyze a LCS. The LCS is analyzed after the calibration standard. The LCS contains a representative subset of the analytes of interest (See Table 8), and must contain the same analytes as the matrix spike. 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. Please refer to Appendix A and Table A-2 for LCS criteria for method 624.

9.6.1 If the batch cannot be re-prepped and/or reanalyzed due to insufficient sample, a discussion should be provided of the data quality indicators and must be clearly presented in the project records and the report.

9.6.2 If re-extraction and/or 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.

9.6.3 Refer to the TestAmerica Pittsburgh QC Program document (PT-QA-021) for further details of the corrective action.

9.6.4 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.

Unless otherwise agreed only the control analytes (Table 8) are used to evaluate analytical performance control.

9.6.5 Use of marginal exceedances are not permitted for South Carolina work.

9.6 Matrix Spikes

For DoD MS/MSD criteria, see SOP # PT-QA-025 and PT-QA-029. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 8. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits. Refer to Table A-2 for method 624 spike limits.

9.6.1 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.

9.6.2 If the recovery for any control component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory operation is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

9.6.3 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.

9.6.4 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.

9.7 Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8 Quality Assurance Summaries

Certain clients may require specific project or program QC, which may supersede these method requirements. Quality Assurance Summaries should be developed by the Project Manager to address these requirements.

9.9 TestAmerica Pittsburgh QC Program

Further details of QC and corrective action guidelines are presented in the TestAmerica Pittsburgh QC Program document (PT-QA-021). Refer to this document if in doubt regarding corrective actions.

10 PROCEDURE

CALIBRATION AND STANDARDIZATION:

10.1 Summary

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 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 seven 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. The use of separate calibrations is required for water and low soil matrices.

10.2 Recommended Instrument Conditions

10.2.1 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

10.2.2 Gas chromatograph suggested temperature program

Parameter	Sample Analysis	BFB Analysis
Initial Temperature:	35□C	35□C
Initial Hold Time:	4 minutes	2 min
Temperature Program:	15□C/minute	20□C/minute
Final Temperature:	200□C	200□C
Final Hold Time:	1.1 minutes	1.0 min.

10.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 9 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.

10.3.1 Acceptable procedures for BFB tuning are as follows:

10.3.1.1 Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex + or - 1 scan and average the three scans. Background correction is required prior to the start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

10.3.1.2 Adjustments such as adjustments to the repeller and the ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as **ALL** of the subsequent injections in the 12 hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than 2 tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in the instrument maintenance log.A single scan at the apex (only) may also be used for the evaluation of the tune. For SW-846 and EPA 600 series methods, background correction is still required.

10.3.1.3 Tune evaluation printouts must include the chromatogram and spectra as well as the tune evaluation information. In addition, the verifications must be sent directly to the printer of pdf file (NO screen prints for BFB tunes). This ability should be built in to the instrument software.

10.3.1.4 If the instrument has a built in macro that checks the BFB, use of this macro with no manual manipulation is also acceptable. (Assuming, of course that the correct ion ratios are being checked.)

10.3.1.5 NOTE: If the background scan selected includes significant ions at 95 or 174 or 176, then the scan is almost certainly part of the BFB peak and is not acceptable.

10.4 Initial Calibration

10.4.1 A series of seven initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Typical calibration levels for a

standard 5 mL purge are: 5, 10, 25, 40, 50, 125 and 250 □g/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Typical calibration levels for a Low Level purge are 1, 5, 10, 15, 20, 35 and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 3 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. 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. See Table 3 and 4 for medium level soil standard concentration. **Note: South Carolina can only be analyzed using linear calibration, quadratic is not allowed.**

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 3) and the Appendix IX standard (Table 4). If acceptable analytical performance can be obtained the primary and appendix IX standards may be analyzed together.

10.4.3 Internal standard calibration is used. The internal standards are listed in Table 6. Target compounds should reference the nearest internal standard (see Table 6A). 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 11 for the CCCs. Acceptable CCC compounds will use average RF curve.

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 10 for the SPCC compounds and required minimum response factors.

10.4.6 Note: the laboratory may not use the "grand mean" rule. The following are guidelines that are used for routine SW-846 analysis within the laboratory, however these guidelines are subject to program and project specific requirements.

10.4.6.1 Where a target compound is ≤15% RSD an average response factor curve may be used. If the 15% RSD criteria are exceeded the

analyst must assess the curve and attempt to apply a "best-fit" curve function and a graphical representation of the curve will be provided as documentation of this review. The first step of the assessment is to find out if the quadratic curve will have a correlation coefficient of ≤ .995. If it does not, then use the average response factor. If it does, then review where the quadratic curve intercepts the y-axis in comparison to the MDL and origin. Also review the shape of the curve. Does it overlap itself or have other potential problems? These steps should all be used in deciding when a quadratic curve or average response factor curve would be best.

10.4.6.2 Where a quadratic or polynomial curve is used R must be ≥.995 for a curve to be considered to be an acceptable fit.

10.4.6.3 All linear curves for non-CCC compounds that exceed 15% RSD or best-fit curve functions that have R < .995 are in exceedance of guidance criteria and must be evaluated for corrective action. The following exceptions may be reportable with narration depending on the project DQO's and data usability requirements:

10.4.6.4 Where a target compound is ≥15% but ≤30% an average response factor curve may still be used if the analyst shows that the average response factor is an acceptable fit over the range of use. A graphical representation of the curve should be presented for documentation. However, if the quadratic curve is clearly a better fit it should be used.

10.4.6.5 Compound list will be divided into two lists: List 1 (reliable performers) and List 2 (poor performers). List 1 compounds should always have a %RSD less than 30% or correlation coefficient of .995 with an allowance of up to two sporadic marginal failures for volatiles. Sporadic marginal failures for these compounds should be </= 40% or .990. Sporadic marginal failures require a print out of the curve and narration.

10.4.6.6 List 2 compounds are comprised of the list of known poor performers. For List 2 analytes, where the %RSD is ≤15% an average response factor will be used. For %RSDs >15% and ≤60% the best fit curve will be selected. For these compounds a print out of the curve will be provided as a graphical documentation of curve performance.

10.4.6.7 Documentation: Raw target curve summary with all compounds set to average response factor will be provided. If quadratic or polynomial equations are used a reprint of the curve table will be

provided to show the correlation coefficient for the "best fit" equation. And as noted above, compounds that need additional documentation to demonstrate the curve fit will have a graphical presentation of the curve provided for reference.

10.4.6.8 Any analyte **not** on List 1 or List 2 would be held to specific criteria based on project specific requirements.

10.4.6.9 Any non-CCC compound being reported from a curve that does not meet either the 15% RSD criteria or the R = .995 for a "best-fit" curve will be narrated as a non-conformance.

10.4.6.10 All %RSDs that are >30% must be narrated and when using an average response factor curve for a %RSD >30% this should also be narrated.

10.4.6.11 **Note:** Project Specific DQOs or program specific requirements supercede routine lab reporting practices listed in this section.

10.4.7 Weighting of data points

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.

10.4.8 If time remains in the 12-hour period initiated by the BFB injection after the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9 A separate seven-point calibration must be prepared for analysis of low level soils. Low level soil analyses require the use of a closed vial autosampler, such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water. 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. Medium soil extracts should be analyzed using the water (unheated) calibration curve.

10.4.10 Non-standard analytes are sometimes requested. Where it is acceptable to the client, it may be is possible to analyze a single standard at the reporting limit (to screen for the compounds) with each continuing calibration rather than a six point initial calibration. If the analyte is detected in any of the samples, a six 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. This is not an acceptable procedure for compliance work. When doing non-standard analytes an MDL will be run before analysis.

10.4.11 All ICALs will be verified by a Second Source Standard. The acceptance criteria are 75-125% for most compounds and 50-150% for poor method performers. The poor performers are footnoted in Tables 3 and 4. Any compound not listed will fall into the 50-150% criteria until knowledge of the compound can be developed. **For DoD QSM 3.0 the second source must be ± 25% for all compounds, refer to SOP PT-QA-025. For DoD QSM 4.1 the second source must be ± 20% for all compounds, refer to SOP PT-QA-029.**

10.4.12 Outliers will be evaluated on a project by project basis and narrated in the case narrative if necessary.

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. The level 3 calibration standard is used as the continuing calibration for low level waters. The level 4 calibration standard is used as the continuing calibration for low level soils and 5 ml waters.

10.5.2 The RF data from the continuing calibration standards are compared with the average RF from the initial seven-point calibration to determine the percent drift or percent deviation of the CCC compounds. The calculations are given in equations 4 (Section 12.3.4) and equation 5 (Section 12.3.5).

10.5.3 Continuing Calibration Verification

10.5.3.1 Calculation Type

10.5.3.1.1 Average Response Factor curves should be verified using a %Difference equation. The %Difference equation compares the RRF factor calculated for the Calibration Verification Standard to the Average RRF of the curve.

10.5.3.1.2 The Quadratic Curves should be verified using a %Drift equation. The %Drift equation compares the measured value of the Calibration Verification Standard to the theoretical value of the standard.

10.5.3.2 %Difference and %Drift Criteria

10.5.3.2.1 CCCs must be ≤20 %Diff

10.5.3.2.2 List One compounds that are non-CCCs must be ≤25 %Diff or Drift

10.5.3.2.3 Up to two Volatile and four Semivolatile compounds that are List One analytes may exceed the 25% criteria, but must be ≤40%.

10.5.3.2.4 List Two Target Analytes including Appendix IX compounds will be accepted where the % Difference or % Drift ≤50%. Please see Table 4-1.

NOTE: See Table 4-2 for South Carolina 8260 ICAL Control List.

10.5.3.2.5 Where a CCV is out high by >50% and the compound is ND in the samples, the samples may be reported with narration.

10.5.3.3 RRF Criteria

10.5.3.3.1 SPCCs must be as per method requirements. Please see Table 10.

10.5.3.3.2 All other compounds must be ≤0.01 (footnote exceptions).

10.5.4 If the CCCs and/or the SPCCs do not meet the criteria in Sections 10.5.3 after the continuing calibration has been attempted twice, 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.).

10.6 Sample Analysis:

10.6.1 Procedural Variations: 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 Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

10.6.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.6.3 See Appendix A for method 624 criteria.

10.6.4 Preliminary Evaluation

10.6.4.1 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.

10.6.4.2 Samples are screened on a headspace analyzer. The instrument is calibrated for select compounds at three levels. There are 200ppb, 500ppb, and 1000ppb. 5 mLs of sample are then analyzed on the headspace analyzer and the results are used to calculate a dilution, if necessary, for the sample.

10.6.4.3 Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or 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 5 µL of sample then serial dilutions must be made in volumetric flasks.

10.6.4.3.1 The diluted concentration is to be estimated to be in the upper half of the calibration range. The upper range will be defined as the mid-range calibration point and above. **NOTE:** TestAmerica Pittsburgh considers a good dilution for

regular waters and soils to be > or = 200 ng on column and for low level waters a good dilution is considered to be > or = 50 ng on column.

10.6.5 Sample Analysis Procedure

10.6.6 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.).

10.6.7 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.

10.6.7.1 If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next 12 hour tune period. However, if any instrument corrective action is required, or if a period of greater than 12 hours (SW-8260B) from the preceding BFB tune has passed, a new batch must be started. In other words a QC batch may be kept open for two adjacent and uninterrupted tune periods where both pass all BFB, CCAL, blank and LCS criteria up to a maximum of 24 hours. For medium level soils the batch is defined at the sample preparation stage. For method 624 the batch tune period is 24 hours.

10.6.7.2 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.

10.6.7.3 It is not necessary to reanalyze batch QC with the reanalyses of samples. However, any reruns must be as part of a valid batch.

10.6.8 For manual integration practices refer to TestAmerica corporate SOP, CA-Q-S-002, Acceptable Manual Integration Practices. For DoD and all other projects the following criteria must be met:

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and name or initials of person performing manual integration operation (electronic signature is acceptable). DoD QSM, Version 3, Clarification 50 and 57.

Case Narrative. For DoD the case narrative shall provide: identification of **samples and analytes** for which manual integration was necessary. DoD QSM, Version 3, Appendix DoD-A and DoD QSM 4.1, Appendix E.

10.6.9 Retention time criteria for samples

Retention time windows must be established and verified once per ICAL and at the beginning of the analytical shift as per DoD QSM, Version 3, Appendix DoD-B, Table B-3 and DoD QSM 4.1 Appendix F, Table F-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.

10.6.9.1 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.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
Retention Time window position establishment for each analyte and surrogate	Once per ICAL	Position shall l be set using the midpoint standard of the initial calibration curve.	NA	NA
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.

10.7 Water Samples

10.7.1 All samples and standard solutions must be at ambient temperature before analysis.

10.7.2 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.

10.7.3 Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 6 and 7). 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 standard 5 mL sample, and a 10 µg/L solution for low level analyses, when added to a 25 mL sample aliquot). Inject the sample into the purging chamber. Note: Low level analyses on instruments that sample directly from the VOA vial (i.e., Archons) use a 5 ml sample volume. Therefore, 1.0 µL of a 250 µg/mL solution of internal standards and surrogates are added to the sample for the regular 5 mL waters and 1uL of a 50 ug/mL solution is added for low level waters.

10.7.3.1 For TCLP samples use 125 uL of TCLP leachate with 4.875 mL reagent water and spike with 8 µL of the 25 µg/mL spiking solution. (Note that TCLP reporting limits will be 40 times higher than the corresponding aqueous limits).

10.7.4 Purge the sample for eleven minutes (the trap must be $\leq$ 35□C).

10.7.5 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

10.7.6 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.

10.8 Methanol Extracted Soils

10.8.1 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 100 µL for a 5 mL purge methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard. Load the sample onto the purge and trap device and analyze the same 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.

10.9 Liquid wastes that are soluble in methanol and insoluble in water.

10.9.1 Pipette 1 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.01 gram. In order to produce an accurate weight to volume relationship take the weight of the liquid sample divided by 1.0 grams to determine a dilution factor which will be applied to reflect this relationship accurately.

10.9.2 Quickly add 8 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD, 7 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

10.9.3 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 100 µL for a 5 mL purge methanolic extract (from Section 11.6.2) to the syringe. Add internal standard. Load the sample onto the purge and trap device and analyze the same 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.

10.10 Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

10.10.1 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.

10.10.2 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 be analyzed as soils.

10.10.3 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.

10.10.4 For aqueous samples, check the pH of the sample remaining in the VOA vial after analysis is completed with narrow range pH paper. If the pH is greater than 2, a nonconformance memo should be initiated.

10.11 Low-Level Solids Analysis using discrete autosamplers:

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 and is not permitted within a number of programs including the PADEP programs.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogates, internal standards, and if applicable, the 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.

10.11.1 Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

10.11.2 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 0.01 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 10.8.

10.11.3 Connect the purge vessel to the purge and trap device.

10.11.4 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 11.8.2.

10.11.5 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

10.11.6 Add the heater jacket or other heating device and start the purge and trap unit.

10.11.7 Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.

10.12 Initial review and corrective actions

10.12.1 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.

10.12.2 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.

10.12.3 Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. Samples that are reported with internal standard exceedances must have documentation supporting matrix effect. Where the matrix effect is well established it may be reported with narration, otherwise the samples must be reanalyzed to confirm matrix effect is required. If the internal standard exceedance is deemed to be due to an instrumental problem, instrument maintenance will be done and all affected samples must be reanalyzed after the problem is corrected.

10.12.4 The surrogate standard recoveries are evaluated to ensure that they are within limits. See section 9.4 for corrective actions for surrogate recoveries.

10.13 Dilutions

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.

10.13.1 Guidance for Dilutions Due to Matrix

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 judgment.

10.13.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11 CALCULATIONS / DATA REDUCTION

11.1 Qualitative identification

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, from the hardcopy printout of the

"clean" reference spectrum book 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.)

11.1.1 The sample component retention time must compare to within at least ±0.06 RRT units of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

11.1.2 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.

11.1.3 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.)

11.1.4 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.

11.1.5 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

11.2 Tentatively Identified Compounds (TICs)

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:

11.2.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.2.2 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%).

11.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.2.5 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.)

11.2.6 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.

11.3 Calculations.

11.3.1 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

Relative Retention Time (RRT) – is the ration of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_C}{RT_{is}}$$

Where,

RT_C = Retention time for the volatile target compounds in the continuing calibration.

RT_{is} = Retention time for the internal standard in calibration standard or in a sample.

11.3.2 Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

11.3.3 Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = **Mean of RF values in the curve**

11.3.4 Percent deviation between the initial calibration and the continuing calibration (%D):

Equation 4

$$\% \text{ Deviation} = \frac{RRFic - RRFcc}{RRFic} \times 100$$

11.3.5 Percent drift between the initial calibration and the continuing calibration:

Equation 5

$$\% \text{ Drift} = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100$$

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

11.3.6 Target compound and surrogate concentrations:

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%.

Calculation of concentration using Average Response Factors

Equation 6

$$\text{Concentration } \mu g / L = \frac{x}{RF}$$

Calculation of concentration using Linear fit

Equation 7

$$\text{Concentration } \mu g / L = A + Bx$$

Calculation of concentration using Quadratic fit

Equation 8

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

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

Calculation of **x** for Water and water-miscible waste:

Equation 9

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

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)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, mL

Calculation of **x** for Medium level soils:

Equation 10

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_x, I_s, D_f, A_{is}, same as for water.

V_t = Volume of total extract, mL

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

$$D = \frac{100 - \%\text{moisture}}{100}$$

Calculation of **x** for Low level soils:

Equation 11

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x, I_s, A_{is}, same as for water.

D is as for medium level soils

W_s = Weight of sample added to the purge vessel, g

Calculation of TICs: The calculation of TICs (tentatively identified compounds) isidentical to the above calculations with the following exceptions:

A_x = Area in 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

In other words, the concentration is equal to **x** as defined in equations 8, 9 and 10.

11.3.7 MS/MSD Recovery and RPD Calculation:

Equation 12

$$\text{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result

SR = Sample result

SA = Spike added

Relative % Difference calculation for the MS/MSD

Equation 13

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

12 METHOD PERFORMANCE

12.1 Method Detection Limit

Generally, the 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 SOP # PT-QA-007. (MDL) studies must be acceptable before analysis of samples may begin. MDLs should be analyzed for low level and soils and aqueous samples. For non-standard analytes, a MDL study or MDL Verification must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method.

12.2 Initial Demonstration

Each laboratory must make a one time 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/LCS 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. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

12.2.2 The performance of all four QC check samples must meet all method requirements for LCSs.

12.2.3 If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of Method 8260B). If the recovery or precision is outside the limits in the reference methods, 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.

12.3 Training Qualification

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 POLLUTION PREVENTION

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2 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."

13.3 This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 PT-HS-001 (or CHP manual). The following waste streams are produced when this method is carried out.

14.1.1 Aqueous waste generated from analysis. This material may have a pH of less than 2.0. This waste is collected in containers identified as “Acid Waste”, Waste #33. It is neutralized to a pH between 6 and 9 and disposed down a lab sink.

14.1.2 Solvent waste generated from analysis. This waste is placed in containers identified as “Vials & Extracts”, Waste #7.

14.1.3 Solid waste generated from analysis. This waste is placed in trash containers and disposed with other building trash.

14.1.4 Expired Standards. This waste is placed in container identified as “Mixed Flammable Solvent Waste”, Waste #3.

15 REFERENCES / CROSS REFERENCES

15.1 SW-846, 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.2 SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, , Revision 2, December 1996.

15.3 SW-846, Method 5030B Purge-And-Trap For Aqueous Samples, Revision 2, December 1996.

15.4 SW-846, Method 5035 Closed-System Purge-And-Trap And Extraction For Volatile Organics In Soil And Waste Samples, Revision 0, December 1996.

15.5 40 CFR Chapter I Part 136, Appendix A, Method 624, 7-1-1997 Edition.

15.6 USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, OSWER 9240.1-05A-P, PG99-963-506, EPA540/R-99/008, October 1999.

15.7 SOP # PT-QA-025, Implementation of DoD QSM Version 3 January 2006, current version.

15.8 SOP # PT-QA-007, Determination of Method Detection Limits (MDL), current version.

15.9 SOP # CA-Q-S-002, Acceptable Manual Integration Practices, current version.

15.10 Pittsburgh Laboratory Quality Assurance Manual, PT-LQAM, current version.

15.11 SOP #PT-QA-029, QA/QC Requirements for DoD QSM 4.1.

15.12 SOP #PT-QA-007, Detection Limits.

15.13 SOP # PT-QA-011, Data Recording Requirements.

15.14 SOP # PT-QA-015, Maintaining Time Integrity.

15.15 SOP #PT-QA-016, Nonconformance An Corrective Action.

15.16 SOP #PT-QA-018, Technical Data Review Requirements.

15.17 SOP # PT-QA-021, Quality Assurance Program.

15.18 SOP #PT-QA-022, Equipment Maintenance.

15.19 SOP #PT-QA-027, Sample Receiving And Chain Of Custody.

16 METHOD MODIFICATIONS

16.1 Modifications from SW-846 Method 8260B

16.1.1 Ion 119 is used as the quantitation ion for chlorobenzene-d5.

16.1.2 A relative retention time window of ±0.06 RT units is used for all components.

16.1.3 The quantitation and qualifier ions for some compounds have been added to the list of those which are recommended in SW-846 in order to improve the reliability of qualitative identification.

16.2 Modification from Method 5035

16.2.1 Presence of residual chlorine is not tested for water samples in section 8.2

16.2.2 Soils samples are not preserved with sodium bisulfate in section 8.4 for low level soils. Refer to sections 8.4 and 8.9.

16.2.3 Flow diagram for Field bisulfate preservation procedure was removed.

17 ATTACHMENTS

17.1 Figure –1 Flow diagram - Initial Demonstration and MDL

17.2 Appendix A, Section 19, Method 624 Requirements

17.3 Tables:

Table 1 - Primary Standard and Reporting Limits for SW846 8260B

Table 2 - Appendix IX Standard and Reporting Limits for SW846 8260B

Table 3 - Primary Standard Calibration Levels

Table 4 - Appendix IX Standard Calibration Levels

Table 4A - Calibration Standard Concentration and Preparation

Table 4-1 – 8260 ICAL Control List

Table 4-2 – 8260 ICAL Control List (South Carolina)

Table 5 - Reportable Analytes for Standard Tests

Table 6 - Internal Standards

Table 6A – Internal Standards with Corresponding Assigned Analytes for Quantitation

Table 7 - Surrogate Standards

Table 8 - Matrix Spike / LCS Standards

Table 9 - BFB Tune Criteria

Table 10 - SPCC Compounds and Minimum Response Factors

Table 11 - CCC Compounds

Table 12 - Characteristic Ions

Table 13 - QC Acceptance Criteria Method 8260B

Table A-1 - Method 624 Analytes and Reporting Limits

Table A-2 - Method 624 QC Acceptance Criteria

Table B-1 - DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3 - DoD QSM Version3: Requirements for Organic Analysis by GC/MS - Methods 8260 and 8270

18 REVISION HISTORY

18.1 Revision 11, 07/14/08:

18.1.1 Updated to new SOP format

18.1.2 Sodium bisulfate removed.

18.1.3 Sections 8.5-8.7, Terra Core added.

18.1.4 Section 9.5 use of sand for Method Blank added.

18.1.5 Section 10.1 calibration standards updated, using 7 levels.

18.1.6 Section 10.3.1, Tune criteria updated to be consistent with Pittsburgh Laboratory Quality Assurance Manual (PT-LQAM).

18.1.7 Section 10.4.11 changed to: The acceptance criteria are 75-125% for most compounds and 50-150% for poor method performers. The poor performers are footnoted in Tables 3 and 4. Any compound not listed will fall into the 50-150% criteria until knowledge of the compound can be developed. **For DoD second source must be ± 25% for all compounds, refer to SOP PT-QA-025.**

18.1.8 Section 10.5 continuing calibration levels updated.

18.1.9 Calibration levels updated, areas are highlighted. Standards tables updated.

18.1.10 SOP and method references updated.

18.2 Revision 12, 11/04/08:

18.2.1 Updated Table 4-1 SPCC designations.

18.2.2 Updated SOP references. Updated Safety section to match Corp SOP format along with sections 13 and 14 Pollution Prevention/Waste Management.

18.3 Revision 13:

18.3.1 Added Section 8.15 concerning Regulatory Requirements for Acrolein, Acrylonitrile and 2-Chloroethyl-vinyl ether in relationship to Holding Times and Preservation.

18.3.2 Added text to section 10.9.1 concerning the application of a dilution factor in accurately reflect the volume to weight relationship.

18.3.3 Updated spike amounts and volumes in section 10.7.3.1 for TCLP.

18.3.4 Removed references and requirements for DoD Version 2.2, this was a typo, Pitt never performed this version.

18.3.5 Updated Table 4-1.

18.3.6 Added to section 9.6.5: Use of marginal exceedances are not permitted for South Carolina work

18.3.7 Added to section 10.4. Note: South Carolina can only be analyzed using linear calibration, quadratic is not allowed.

18.3.8 Added references to DoD QSM 4.1, SOP PT-QA-029.

18.4 Revision 14

18.4.1 In section 7.2.6 corrected the unit to read ng/mL.

18.4.2 In section 10.4.3 added reference to Table 6A – Internal Standards with Corresponding Assigned Analytes for Quantitation.

18.4.3 In section 10.4.11 corrected grammar from criteria is to criteria are.

18.4.4 Under section 10.5.3.2.4 added reference to Table 4-2 8260 ICAL Control List (South Carolina). Footnote 3 under Table 4-2 had the following statement added: The most common poor purging List 1 compounds are Carbon tetrachloride, cis-1,3-Dichloropropene and trans-1,3-Dichloropropene.

18.4.5 In section 17, added reference to Table 4-1 8260 ICAL Control List; Table 4-2 8260 ICAL Control List (South Carolina); Table 6A – Internal Standards with Corresponding Assigned Analytes for Quantitation.

18.4.6 Added a **NOTE** in section 19.4.1 to clarify the CCAL concentration when analyzing 8260B and 624 samples together. Noted that a 10 ug/L standard will be used for calibration instead of a 20 ug/L and that all criteria for both Methods must be met in order to analyze samples.

18.4.7 Removed reference to OVAP in sections 8.9.1, 8.10.2 and 10.11.

18.4.8 Removed the DoD QSM 3.0 tables and added them to DoD QSM 3.0 SOP PT-QA-025.

18.4.9 Added reference to DoD QSM 4.1 and SOP PT-QA-029 in section 1.2

18.4.10 In section 1.4 changed 200 ug/L to 250 ug/L as per the VOA group.

18.4.11 Specified the ± 20% DoD QSM 4.1 criteria for the ICV in section 10.4.11.

18.4.12 In section 10.6.8 added reference to DoD QSM 4.1 Appendix E.

18.4.13 In section 10.6.9 added reference to DoD QSM 4.1 Appendix F, Table F-1.

18.4.14 In section 10.6.4.3.1 changed 250 ng to 200 ng as per the VOA group.

18.5 Revision 15:

18.5.1 Fixed Typos for two compounds in Tables 4-1 and 4-2: 1,1,2-Trichloro-1,2,2-Trifluoroethane and 1,2-Dibromoethane.

18.5.2 Deleted SPCC and Min FR 0.1 for ,1,2-Dichloroethane in Tables 4-1 and 4-2.

18.5.3 Updated foot note in Table 4-2 to meet SC requirements: List 2 compounds – All compounds must meet 70-130% with the exception of the identified poor purging compounds which are identified as Carbon tetrachloride, cis-1,3-Dichloropropene and trans-1,3-Dichloropropene. Any compounds outside of the 70-130% range (0r 40% for poor purgers) must be flagged in the data and narrated.

18.5.4 Added SOP references.

Figure 1 - Flow diagram - Initial Demonstration and MDL

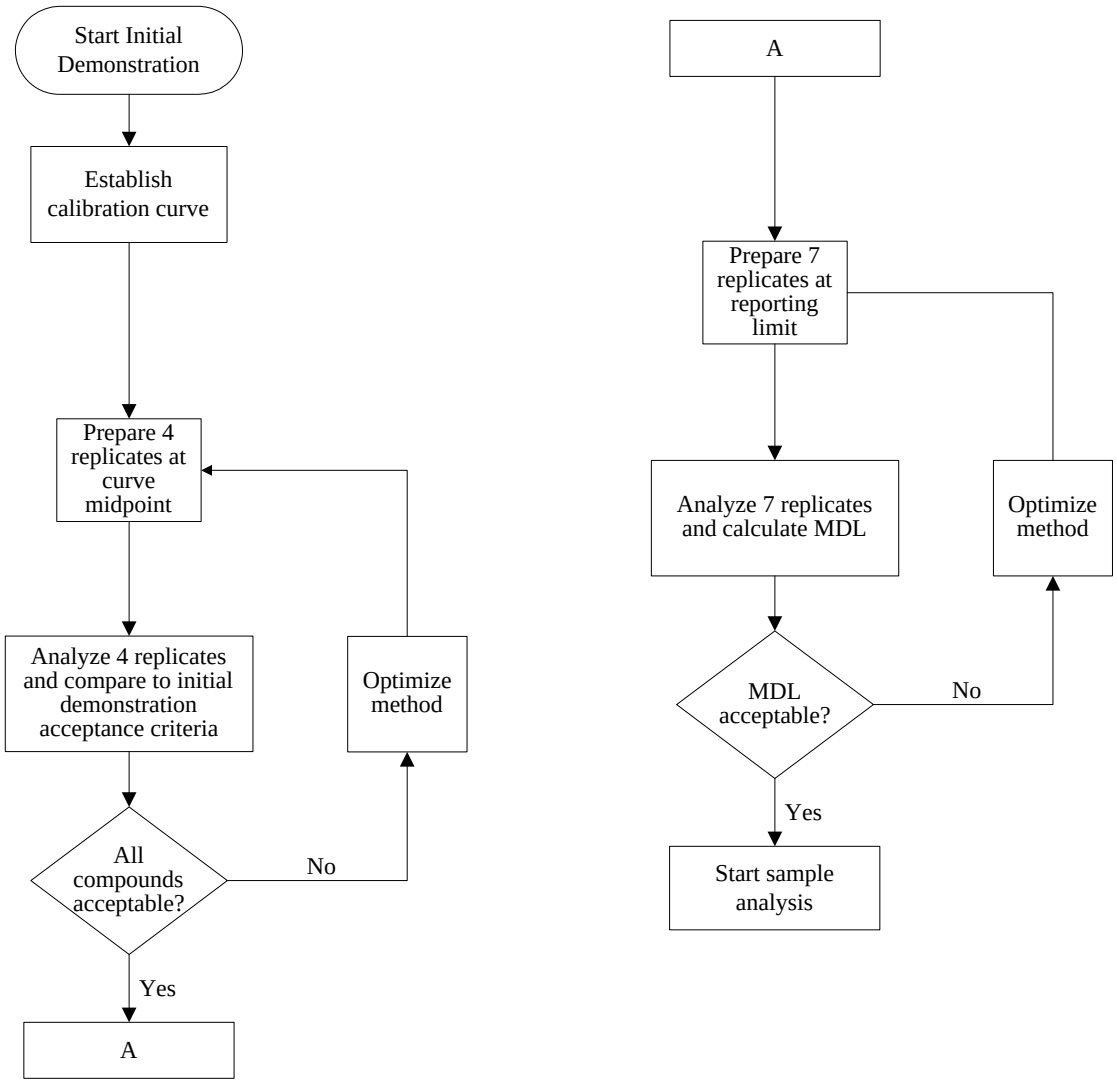


Table 1
Primary Standard and Reporting Limits for SW846 8260B

Compound	CAS Number	Low Level water µg/L	5 mL Water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	1	5	5	250
Chloromethane	74-87-3	1	5	5	250
Bromomethane	74-83-9	1	5	5	250
Vinyl chloride	75-01-4	1	5	5	250
Chloroethane	75-00-3	1	5	5	250
Trichlorofluoromethane	75-69-4	1	5	5	250
Acetone	67-64-1	10	20	20	1000
Trichlorotrifluoroethane	76-13-1	1	5	5	250
Carbon disulfide	75-15-0	1	5	5	250
Methylene chloride	75-09-2	1	5	5	250
1,1-Dichloroethene	75-35-4	1	5	5	250
1,1-Dichloroethane	75-34-3	1	5	5	250
trans-1,2-Dichloroethene	156-60-5	1	5	5	250
Methyl tert-butyl ether (MTBE)	1634-04-4	1	5	5	250
cis-1,2-Dichloroethene	156-59-2	1	5	5	250
1,2-Dichloroethene (Total)	540-59-0	1	5	5	250
Chloroform	67-66-3	1	5	5	250
1,2-Dichloroethane	107-06-2	1	5	5	250
Dibromomethane	74-95-3	1	5	5	250
2-Butanone	78-93-3	5	20	20	1000
1,1,1-Trichloroethane	71-55-6	1	5	5	250
Carbon tetrachloride	56-23-5	1	5	5	250
Bromodichloromethane	75-27-4	1	5	5	250
1,2-Dichloropropane	78-87-5	1	5	5	250
cis-1,3-Dichloropropene	10061-01-5	1	5	5	250
Trichloroethene	79-01-6	1	5	5	250
Dibromochloromethane	124-48-1	1	5	5	250
1,2-Dibromoethane	106-93-4	1	5	5	250
1,2,3-Trichloropropane	96-18-4	1	5	5	250
1,1,2-Trichloroethane	79-00-5	1	5	5	250
Benzene	71-43-2	1	5	5	250
trans-1,3-Dichloropropene	10061-02-6	1	5	5	250
Bromoform	75-25-2	1	5	5	250
4-Methyl-2-pentanone	108-10-1	5	20	20	1000
2-Hexanone	591-78-6	5	20	20	1000
Tetrachloroethene	127-18-4	1	5	5	250
Toluene	108-88-3	1	5	5	250
1,1,2,2-Tetrachloroethane	79-34-5	1	5	5	250

Table 1
Primary Standard and Reporting Limits for SW846 8260B

Compound	CAS Number	Low Level water µg/L	5 mL Water µg/L	Low soil µg/kg	Med. Soil µg/kg
1,1,1,2-Tetrachloroethane	630-20-6	1	5	5	250
1,2-Dibromo-3-chloropropane	96-12-8	1	5	5	250
Chlorobenzene	108-90-7	1	5	5	250
Ethylbenzene	100-41-4	1	5	5	250
Styrene	100-42-5	1	5	5	250
m and p Xylenes		2	10	10	500
o-xylene	95-47-6	1	5	5	250
Total xylenes	1330-20-7	3	15	15	750
p-Isopropyltoluene	99-87-6	1	5	5	250
Methylcyclohexane	108-87-2	1	5	5	250
1,1,2-Trichloro-1,2,2-Trifluoroethane	76-13-1	1	5	5	250
Methyl acetate	79-20-9	1	5	5	250
Cyclohexane	110-82-7	1	5	5	250
1,3-Dichlorobenzene	541-73-1	1	5	5	250
1,4-Dichlorobenzene	106-46-7	1	5	5	250
1,2-Dichlorobenzene	95-50-1	1	5	5	250
Isopropylbenzene	98-82-8	1	5	5	250
Bromobenzene	108-86-1	1	5	5	250
n-Propylbenzene	103-65-1	1	5	5	250
2-Chlorotoluene	95-49-8	1	5	5	250
4-Chlorotoluene	106-43-4	1	5	5	250
1,3,5-Trimethylbenzene	108-67-8	1	5	5	250
tert-Butylbenzene	98-06-6	1	5	5	250
1,2,4-Trimethylbenzene	95-63-6	1	5	5	250
sec-Butylbenzene	135-98-8	1	5	5	250
n-Butylbenzene	104-51-8	1	5	5	250
1,2,4-Trichlorobenzene	120-82-1	1	5	5	250
Naphthalene	91-20-3	1	5	5	250
Hexachlorobutadiene	87-68-3	1	5	5	250
1,2,3-Trichlorobenzene	87-61-6	1	5	5	250

[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.

Table 2
Appendix IX Standard and Reporting Limits for SW846 8260B

Compound	CAS Number	Low level water µg/L	5 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	1	5	5	250
Acetonitrile	75-05-8	20	100	100	5000
Acrolein	107-02-8	20	100	100	5000
Chloroprene	126-99-8	1	5	5	250
Iodomethane	74-88-4	1	5	5	250
Propionitrile	107-12-0	2	10	10	500
Methacrylonitrile	126-98-7	1	5	5	250
Isobutanol	78-83-1	40	200	200	10000
Iodomethane	74-88-4	1	5	5	250
Methyl methacrylate	80-62-6	1	5	5	250
Acrylonitrile	107-13-1	20	100	100	5000
Ethylmethacrylate	97-63-2	1	5	5	250
2-Chloroethyl vinyl ether[1]	110-75-8	2	10	10	500
tert-Butyl Alcohol	75-65-0	40	200	200	10,000
1,4-Dioxane	123-91-1	200	1000	1000	50000
Vinyl acetate	108-05-4	1	5	5	250
t-1,4-Dichloro-2-butene	110-57-6	1	5	5	250

Table 3

Primary Standard Calibration Levels, Standard 5 mL purge (Low Level Calibration Levels)

Compound	Calibration Level (ug/L)						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,2-Dichloroethane-d4 (Surrogate)	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Toluene-d8 (Surrogate)	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
4-Bromofluorobenzene (Surrogate)	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Dichlorodifluoromethane *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Chloromethane *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Bromomethane *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Vinyl chloride *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Chloroethane *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Trichlorofluoromethane *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Acetone *	5 (2)	10 (10)	25 (10)	40 (30)	50 (40)	125 (70)	250 (80)
Carbon disulfide *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Methylene chloride	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Isopropylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,1-Dichloroethene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,1-Dichloroethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
trans-1,2-Dichloroethene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,1,1,2-Tetrachloroethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Methyl tert-butyl ether (MTBE) *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2-Dibromo-3-chloropropane *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
cis-1,2-Dichloroethene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Chloroform	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2-Dichloroethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Dibromomethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
2-Butanone *	5 (2)	10 (10)	25 (20)	40 (15)	50 (40)	125 (70)	250 (80)
1,1,1-Trichloroethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Carbon tetrachloride	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Bromodichloromethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2-Dichloropropane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
cis-1,3-Dichloropropene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Trichloroethene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Dibromochloromethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2-Dibromoethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2,3-Trichloropropane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,1,2-Trichloroethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Benzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
trans-1,3-Dichloropropene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)

Table 3

Primary Standard Calibration Levels, Standard 5 mL purge (Low Level Calibration Levels)

Compound	Calibration Level (ug/L)						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Bromoform	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
4-Methyl-2-pentanone *	5 (2)	10 (10)	25 (20)	40 (30)	50 (40)	125 (70)	250 (80)
2-Hexanone *	5 (2)	10 (10)	25 (20)	40 (30)	50 (40)	125 (70)	250 (80)
Tetrachloroethene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Toluene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,1,2,2-Tetrachloroethane	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Chlorobenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Ethylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Styrene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
m and p Xylenes	10 (2)	20 (10)	50 (20)	80 (30)	100 (40)	125 (70)	500 (80)
o-xylene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,3-Dichlorobenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,4-Dichlorobenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2-Dichlorobenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Isopropylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Bromobenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
n-Propylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
2-Chlorotoluene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
4-Chlorotoluene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,3,5-Trimethylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
tert-Butylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2,4-Trimethylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
sec-Butylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
n-Butylbenzene	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2,4-Trichlorobenzene *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Naphthalene *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Hexachlorobutadiene *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
1,2,3-Trichlorobenzene *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)

For medium level soils the above standard concentrations will be multiplied by 50.

Table 4
Appendix IX Standard Calibration Levels, Standard 5 mL purge (Low Level Calibration Levels)

Compound	Calibration Level (ug/L)						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Allyl Chloride *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Acetonitrile *	100 (20)	200 (100)	500 (200)	800 (300)	1000 (400)	2500 (700)	5000 (800)
Chloroprene *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Propionitrile *	10 (2)	20 (10)	50 (20)	80 (30)	100 (40)	250 (70)	500 (80)
Methacrylonitrile *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	125 (35)	250 (40)
Isobutanol *	200 (40)	400 (200)	1000 (400)	1600 (600)	2000 (800)	5000 (1400)	10000 (1600)
Methyl methacrylate *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	250 (35)	50 (40)
Acrolein *	100 (20)	125 (25)	150 (30)	175 (35)	200 (40)	225 (45)	250 (50)
1,4-Dioxane *	1000 (200)	2000 (1000)	5000(2000)	8000 (3000)	10000 (4000)	25000 (7000)	50000 (8000)
tert-Butyl alcohol *	200 (40)	400 (200)	1000 (400)	1600 (600)	2000 (800)	5000 (1400)	10000 (1600)
Acrylonitrile *	100 (20)	125 (25)	150 (30)	175 (35)	200 (40)	225 (45)	250 (50)
Ethylmethacrylate *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	250 (35)	50 (40)
2-Chloroethyl vinyl ether*	10 (2)	20 (10)	50 (20)	80 (30)	100 (40)	250 (70)	500 (80)
Vinyl Acetate *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	250 (35)	50 (40)
t-1,4-Dichloro-2-butene*	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	250 (35)	50 (40)
Iodomethane *	5 (1)	10 (5)	25 (10)	40 (15)	50 (20)	250 (35)	50 (40)

* Poor method performers (see section 10.4.11)

For medium level soils the above standard concentrations will be multiplied by 50.

Table 4A – Calibration Standard Concentration and Preparation

Standard Level 8260B Water or Soil 5mL syringe

STD	INT (25ug/mL)	SURR (25ug/mL)	VOA (25ug/mL)	Acetonitrile (1000ug/mL)	Methanol Added
5ppb	10ul	1ul	1ul	0.5ul	122.5ul
10ppb	10ul	2ul	2ul	1ul	120ul
25ppb	10ul	5ul	5ul	2.5ul	112.5ul
40ppb	10ul	8ul	8ul	4ul	105ul
50ppb	10ul	10ul	10ul	5ul	100ul
125ppb	10ul	25ul	25ul	12.5ul	62.5ul
250ppb	10ul	50ul	50ul	25ul	0

8260B App IX Water/Soil 5mL syringe

STD	INT 25ug/mL	App IX 25ug/mL	2CEVE 50ug/mL	A&A 25ug/mL	TBA 1000ug/mL	n-Heptane 25ug/mL
5ppb	10ul	1ul	1ul	20ul	1ul	1ul
10ppb	10ul	2ul	2ul	25ul	2ul	2ul
25ppb	10ul	5ul	5ul	30ul	5ul	5ul
40ppb	10ul	8ul	8ul	35ul	8ul	8ul
50ppb	10ul	10ul	10ul	40ul	10ul	10ul
125ppb	10ul	25ul	25ul	45ul	25ul	25ul
250ppb	10ul	50ul	50ul	50ul	50ul	50ul

Table 4A – Calibration Standard Concentration and Preparation Cont.

Dupont 5mL syringe

STD	INT 25ug/mL	Dupont VOA 25ug/mL	Dupont Acrylates 25ug/mL
5ppb	10ul	1ul	1ul
10ppb	10ul	2ul	2ul
25ppb	10ul	5ul	5ul
40ppb	10ul	8ul	8ul
50ppb	10ul	10ul	10ul
125ppb	10ul	25ul	25ul
250ppb	10ul	50ul	50ul

CLP OLM04.1/3.1/3.2 Water & Soil 5mL syringe

STD	INT (25ug/mL)	SURR (25ug/mL)	VOA (25ug/mL)	Methanol Added
10ppb	10ul	2ul	2ul	80ul
20ppb	10ul	4ul	4ul	70ul
50ppb	10ul	10ul	10ul	60ul
100ppb	10ul	20ul	20ul	40ul
200ppb	10ul	40ul	40ul	0

Table 4A – Calibration Standard Concentration & Preparation Cont.

624 & Low Level 8260B Water 25mL syringe

STD	INT 25ug/mL	SURR (25ug/mL)	VOA (25ug/mL)	Acetonitrile (1000ug/mL)	Ketone 25ug/mL	Methanol Added
1ppb	10ul	1ul	1ul	0.5ul	4ul	140ul
5ppb	10ul	5ul	5ul	2.5ul	5ul	120ul
10ppb	10ul	10ul	10ul	5.0ul	10ul	105ul
15ppb	10ul	15ul	15ul	7.5ul	15ul	88ul
20ppb	10ul	20ul	20ul	10ul	20ul	70ul
35ppb	10ul	35ul	35ul	17.5ul	35ul	17.5ul
40ppb	10ul	40ul	40ul	20ul	40ul	0

8260B Low Level App IX Water 25mL syringe

STD	INT 25ug/mL	App IX (Various)	2CEVE 50ug/mL	A&A 25ug/mL	TBA 1000ug/mL
1ppb	10ul	1ul	1ul	20ul	1ul
5ppb	10ul	5ul	5ul	25ul	5ul
10ppb	10ul	10ul	10ul	30ul	10ul
15ppb	10ul	15ul	15ul	35ul	15ul
20ppb	10ul	20ul	20ul	40ul	20ul
35ppb	10ul	35ul	35ul	45ul	45ul
40ppb	10ul	40ul	40ul	50ul	50ul

Table 4-1
8260 ICAL Control List

Compound	SW-846	Control	SPCC	Comments
1,1-Dichloroethene	8260B	CCC [1]		
Chloroform	8260B	CCC		
Ethylbenzene	8260B	CCC		
Toluene	8260B	CCC		
Vinyl Chloride	8260B	CCC		
1,2-Dichloropropane	8260B	CCC		
1,1,1-Trichloroethane	8260B	1		
1,1,2,2-Tetrachloroethane	8260B	SPCC	SPCC[3]	Min RF 0.3
1,1,2-Trichloroethane	8260B	1		
1,1-Dichloroethane	8260B	SPCC		Min RF 0.1
1,2-Dichlorobenzene	8260B	1		
1,2-Dichloroethane	8260B	1		
1,3-Dichlorobenzene	8260B	1		
1,4-Dichlorobenzene	8260B	1		
Benzene	8260B	1		
Bromodichloromethane	8260B	1		
Bromoform	8260B	SPCC	SPCC	Min RF 0.1
Bromomethane (Methyl Bromide)	8260B	2		
Carbon Tetrachloride	8260B	1		
Chlorobenzene	8260B	SPCC	SPCC	Min RF 0.3
Cis-1,3-Dichloropropene	8260B	1		
Dibromochloromethane	8260B	1		
Styrene	8260B	1		
Tetrachloroethene	8260B	1		
Trans-1,3-Dichloropropene	8260B	1		
Trichloroethene	8260B	1		
Xylenes (total)	8260B	2		
1,2,4-Trichlorobenzene	8260B	2		
1,1,2-Trichloro-1,2,2-Trifluoroethane	8260B	2[4]		
1,2-Dibromo-3-Chloropropane	8260B	2		
1,2-Dibromoethane	8260B	2		
1,2-Dichloroethene (total)	8260B	2		
2-Butanone (MEK)	8260B	2		
2-Hexanone	8260B	2		
4-Methyl-2-Pentanone	8260B	2		
Acetone	8260B	2		

Table 4-1
8260 ICAL Control List

Compound	SW-846	Control	SPCC	Comments
Carbon Disulfide	8260B	2		
Chloroethane	8260B	2		
Chloromethane (Methyl Chloride)	8260B	SPCC	SPCC	Min RF 0.1
Cis-1,2-Dichloroethene	8260B	2		
Cyclohexane	8260B	2		
Dichlorodifluoromethane	8260B	2		
Isopropylbenzene	8260B	2		
Methyl Acetate	8260B	2		
Methyl Tert-Butyl Ether (MTBE)	8260B	2		
Methylcyclohexane	8260B	2		
Methylene Chloride	8260B	2		
Trans-1,2-Dichloroethene	8260B	2		
Trichlorofluoromethane	8260B	2		
1,1,1,2-Tetrachloroethane	8260B	2		
1,2-Dichloropropene	8260B	2		
1,2,3-Trichlorobenzene	8260B	2		
1,2,3-Trichloropropane	8260B	2		
1,2,4-Trimethylbenzene	8260B	2		
1,2-Dibromoethane (EDB)	8260B	2		
1,3,5-Trimethylbenzene	8260B	2		
1,3-Dichloropropane	8260B	2		
1,4-Dioxane	8260B	2		APPIX
2,2-Dichloropropane	8260B	2		
2-Butanone (MEK)	8260B	2		
2-Chloroethyl Vinyl Ether	8260B	2		APPIX
2-Chlorotoluene	8260B	2		
4-Chlorotoluene	8260B	2		
4-Methyl-2-Pentanone (MIBK)	8260B	2		
Acetonitrile	8260B	2		APPIX
Acrolein	8260B	2		APPIX
Acrylonitrile	8260B	2		APPIX
Ally Chloride	8260B	2		APPIX
Bromobenzene	8260B	2		
Bromochloromethane	8260B	2		
Chlorodibromomethane	8260B	2		
Chloroprene	8260B	2		APPIX
Dibromomethane (Methylene Bromide)	8260B	2		

Table 4-1
8260 ICAL Control List

Compound	SW-846	Control	SPCC	Comments
Dichlorobromomethane	8260B	2		
Ethyl Methacrylate	8260B	2		APPIX
Hexachlorobutadiene	8260B	2		
Iodomethane (Methyl Iodide)	8260B	2		APPIX
Isobutanol	8260B	2		APPIX
Isobutyl Alcohol	8260B	2		APPIX
m-Dichlorobenzene	8260B	2		
Methacrylonitrile	8260B	2		APPIX
Methyl Methacrylate	8260B	2		
m-Xylene & p-Xylene	8260B	2		
Naphthalene	8260B	2		
n-Butylbenzene	8260B	2		
n-Propylbenzene	8260B	2		
o-Dichlorobenzene	8260B	2		
o-Xylene	8260B	2		
p-Dichlorobenzene	8260B	2		
p-Isopropyltoluene	8260B	2		
Propionitrile	8260B	2		APPIX
Sec-Butylbenzene	8260B	2		
Tert-Butylbenzene	8260B	2		
Tetrachloroethene	8260B	2		
Traas-1,4-Dichloro-2-butene	8260B	2		APPIX
Vinyl Acetate	8260B	2		APPIX

[1] CCC'S Must Be <20% No Exceptions

[2] SPCC's Must Pass Minimum RF Requirements

[3] List 1 Can Have Up To 2 Compounds Above 25%D But Must Be <40%.

[4] List 2 Compounds Can Be Over 40%D, However If The %D Is >50% (Too High) It Can Be Narrated As Long As The Compound(S) Are ND In The Samples; If The Compounds >50%D (Too Low) This Compound Cannot Be Analyzed For On That Particular CCAL.

Narrative Issues:

- All %RSD that >30% must be narrated. This may be changed with the development of a calibration summary sheet.
- All %Diff or %Drift >25% must be narrated.
- Any other criteria exceedance aside from these should be narrated.
- Using an average response factor curve for a %RDS ≥30% should be narrated.

- If a list two compound > 50% D or Drift and is out high and this compound is not found in the associated samples it may be reported with narration.
  Note: These criterion are subject to project-specific criteria which may vary depending on project needs.

Table 4-2
8260 ICAL Control List (South Carolina)

Compound	SW-846	Control	SPCC	Comments
1,1-Dichloroethene	8260B	CCC [1]		
Chloroform	8260B	CCC		
Ethylbenzene	8260B	CCC		
Toluene	8260B	CCC		
Vinyl Chloride	8260B	CCC		
1,2-Dichloropropane	8260B	CCC		
1,1,1-Trichloroethane	8260B	1		
1,1,2,2-Tetrachloroethane	8260B	SPCC	SPCC[3]	Min RF 0.3
1,1,2-Trichloroethane	8260B	1		
1,1-Dichloroethane	8260B	SPCC		Min RF 0.1
1,2-Dichlorobenzene	8260B	1		
1,2-Dichloroethane	8260B	1		
1,3-Dichlorobenzene	8260B	1		
1,4-Dichlorobenzene	8260B	1		
Benzene	8260B	1		
Bromodichloromethane	8260B	1		
Bromoform	8260B	SPCC	SPCC	Min RF 0.1
Bromomethane (Methyl Bromide)	8260B	2		
Carbon Tetrachloride	8260B	1		
Chlorobenzene	8260B	SPCC	SPCC	Min RF 0.3
Cis-1,3-Dichloropropene	8260B	1		
Dibromochloromethane	8260B	1		
Styrene	8260B	1		
Tetrachloroethene	8260B	1		
Trans-1,3-Dichloropropene	8260B	1		
Trichloroethene	8260B	1		
Xylenes (total)	8260B	2		
1,2,4-Trichlorobenzene	8260B	2		
1,1,2-Trichloro-1,2,2-Trifluoroethane	8260B	2[4]		
1,2-Dibromo-3-Chloropropane	8260B	2		
1,2-Dibromoethane	8260B	1		
1,2-Dichloroethene (total)	8260B	1		
2-Butanone (MEK)	8260B	2		

Table 4-2
8260 ICAL Control List (South Carolina)

Compound	SW-846	Control	SPCC	Comments
2-Hexanone	8260B	2		
4-Methyl-2-Pentanone	8260B	2		
Acetone	8260B	2		
Carbon Disulfide	8260B	2		
Chloroethane	8260B	2		
Chloromethane (Methyl Chloride)	8260B	SPCC	SPCC	Min RF 0.1
Cis-1,2-Dichloroethene	8260B	1		
Cyclohexane	8260B	1		
Dichlorodifluoromethane	8260B	2		
Isopropylbenzene	8260B	1		
Methyl Acetate	8260B	2		
Methyl Tert-Butyl Ether (MTBE)	8260B	1		
Methylcyclohexane	8260B	2		
Methylene Chloride	8260B	1		
Trans-1,2-Dichloroethene	8260B	1		
Trichlorofluoromethane	8260B	2		
1,1,1,2-Tetrachloroethane	8260B	2		
1,2-Dichloropropene	8260B	1		
1,2,3-Trichlorobenzene	8260B	2		
1,2,3-Trichloropropane	8260B	1		
1,2,4-Trimethylbenzene	8260B	1		
1,2-Dibromoethane (EDB)	8260B	1		
1,3,5-Trimethylbenzene	8260B	1		
1,3-Dichloropropane	8260B	1		
1,4-Dioxane	8260B	--		APPIX
2,2-Dichloropropane	8260B	2		
2-Butanone (MEK)	8260B	2		
2-Chloroethyl Vinyl Ether	8260B	--		APPIX
2-Chlorotoluene	8260B	1		
4-Chlorotoluene	8260B	1		
4-Methyl-2-Pentanone (MIBK)	8260B	2		
Acetonitrile	8260B	--		APPIX
Acrolein	8260B	--		APPIX
Acrylonitrile	8260B	--		APPIX
Ally Chloride	8260B	--		APPIX
Bromobenzene	8260B	2		
Bromochloromethane	8260B	2		

Table 4-2
8260 ICAL Control List (South Carolina)

Compound	SW-846	Control	SPCC	Comments
Chlorodibromomethane	8260B	2		
Chloroprene	8260B	--		APPIX
Dibromomethane (Methylene Bromide)	8260B	1		
Dichlorobromomethane	8260B	1		
Ethyl Methacrylate	8260B	--		APPIX
Hexachlorobutadiene	8260B	2		
Iodomethane (Methyl Iodide)	8260B	--		APPIX
Isobutanol	8260B	--		APPIX
Isobutyl Alcohol	8260B	--		APPIX
m-Dichlorobenzene	8260B	1		
Methacrylonitrile	8260B	--		APPIX
Methyl Methacrylate	8260B	2		
m-Xylene & p-Xylene	8260B	2		
Naphthalene	8260B	2		
n-Butylbenzene	8260B	2		
n-Propylbenzene	8260B	2		
o-Dichlorobenzene	8260B	1		
o-Xylene	8260B	2		
p-Dichlorobenzene	8260B	1		
p-Isopropyltoluene	8260B	1		
Propionitrile	8260B	--		APPIX
Sec-Butylbenzene	8260B	2		
Tert-Butylbenzene	8260B	2		
Tetrachloroethene	8260B	1		
Trans-1,4-Dichloro-2-butene	8260B	--		APPIX
Vinyl Acetate	8260B	--		APPIX

[1] CCC'S Must Be <20% No Exceptions

[2] SPCC's Must Pass Minimum RF Requirements

[3] List 1 Can Have Up To 2 Compounds Above 25%D But Must Be <40%. The most common poor purging List 1 compounds are Carbon tetrachloride, cis-1,3-Dichloropropene and trans-1,3-Dichloropropene.

[4] List 2 compounds – All compounds must meet 70-130% with the exception of the identified poor purging compounds which are identified as Carbon tetrachloride, cis-1,3-Dichloropropene and trans-1,3-Dichloropropene. Any compounds outside of the 70-130% range (0r 40% for poor purgers) must be flagged in the data and narrated.

Narrative Issues:

- All %RSD that >30% must be narrated. This may be changed with the development of a calibration summary sheet.
- All %Diff or %Drift >25% must be narrated.
- Any other criteria exceedance aside from these should be narrated.
- Using an average response factor curve for a %RDS ≥30% should be narrated.
- If a list two compound > 50% D or Drift and is out high and this compound is not found in the associated samples it may be reported with narration.

  Note: These criterion are subject to project-specific criteria which may vary depending on project needs.

Table 5
Reportable Analytes for TestAmerica Standard Tests

Compound	CAS Number	624	8260	Appendix IX	CLP 4.2
Dichlorodifluoromethane	75-71-8		X	X	X
Chloromethane	74-87-3	X	X	X	X
Bromomethane	74-83-9	X	X	X	X
Vinyl chloride	75-01-4	X	X	X	X
Chloroethane	75-00-3	X	X	X	X
Trichlorofluoromethane	75-69-4	X	X	X	X
Acrolein	107-02-8			X	
Acetone	67-64-1		X	X	X
Iodomethane	74-88-4			X	
Carbon disulfide	75-15-0		X	X	X
Methylene chloride	75-09-2	X	X	X	X
tert-Butyl alcohol	75-65-0			X	
1,1-Dichloroethene	75-35-4	X	X	X	X
1,1-Dichloroethane	75-34-3	X	X	X	X
trans-1,2-Dichloroethene	156-60-5	X	X	X	X
Acrylonitrile	107-13-1			X	
Methyl tert-butyl ether (MTBE)	1634-04-4	X	X	X	X
cis-1,2-Dichloroethene	156-59-2		X	X	X
Chloroform	67-66-3	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X
Dibromomethane	74-95-3		X	X	
2-Butanone	78-93-3		X	X	X
1,4-Dioxane	123-91-1			X	
1,1,1-Trichloroethane	71-55-6	X	X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X
Bromodichloromethane	75-27-4	X	X	X	X
1,2-Dichloropropane	78-87-5	X	X	X	X
cis-1,3-Dichloropropene	10061-01-5	X	X	X	X
Trichloroethene	79-01-6	X	X	X	X
Dibromochloromethane	124-48-1	X	X	X	X
1,2-Dibromoethane	106-93-4		X	X	X
1,2,3-Trichloropropane	96-18-4		X	X	
1,1,2-Trichloroethane	79-00-5	X	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	X

Table 5
Reportable Analytes for TestAmerica Standard Tests

Compound	CAS Number	624	8260	Appendix IX	CLP 4.2
Bromoform	75-25-2	X	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	X
1,1,2,2-Tetrachloroethane	79-34-5	X	X	X	X
2-Chloroethyl vinyl ether	110-75-8	X		X	
Vinyl acetate	108-05-4			X	
Chlorobenzene	108-90-7	X	X	X	X
Ethylbenzene	100-41-4	X	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	
o-xylene	95-47-6		X	X	
Total xylenes	1330-20-7		X	X	X
1,3-Dichlorobenzene	541-73-1	X	X		X
1,4-Dichlorobenzene	106-46-7	X	X		X
1,2-Dichlorobenzene	95-50-1	X	X		X
1,2-Dichloroethene (total)	540-59-0		X		
2,2-Dichloropropane	590-20-7		X	X	
Bromochloromethane	74-97-5		X		
1,1-Dichloropropene	563-58-6		X		
1,3-Dichloropropane	142-28-9		X		
1,1,1,2-Tetrachloroethane	630-20-6		X	X	
Isopropylbenzene	98-82-8		X		X
Bromobenzene	108-86-1		X		
n-Propylbenzene	103-65-1		X		
2-Chlorotoluene	95-49-8		X		
4-Chlorotoluene	106-43-4		X		
1,3,5-Trimethylbenzene	108-67-8		X		
tert-Butylbenzene	98-06-6		X		
1,2,4-Trimethylbenzene	95-63-6		X		
sec-butylbenzene	135-98-8		X		
4-Isopropyltoluene	99-87-6		X		
n-Butylbenzene	104-51-8		X		
1,2-Dibromo-3-chloropropane	96-12-8		X		X
1,2,4-Trichlorobenzene	120-82-1		X		X

Table 5
Reportable Analytes for TestAmerica Standard Tests

Compound	CAS Number	624	8260	Appendix IX	CLP 4.2
Napthalene	91-20-3		X		
Hexachlorobutadiene	87-68-3		X		
1,2,3-Trichlorobenzene	87-61-6		X		
Methylcyclohexane	108-87-2		X		X
1,1,2-Trichloro-1,2,2-Triflroroethane	76-13-1		X		X
Methyl Acetate	79-20-9		X		X
Allyl Chloride	107-05-1			X	
Acetonitrile	75-05-8			X	
Chloroprene	126-99-8			X	
Propionitrile	107-12-0			X	
Methacrylonitrile	126-98-7			X	
Isobutanol	78-83-1			X	
Methyl methacrylate	80-62-6			X	

Table 6
Internal Standards

Internal Standard Compound	Standard Concentration µg/mL	Quantitation ion (m/z)
Fluorobenzene	25	96
Chlorobenzene-d5	25	119
1,4-Dichlorobenzene-d4	25	152

Notes:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a standard 5 mL purge Method 8260B, or 10 µg/L for low level Method 8260B waters (which uses a 25 ml sample aliquot), Method 624. For instruments that sample directly from the VOA vial, 10 µL of a 5 µg/mL internal standard solution is added to low level Method 8260B waters, and Method 624 since the instrument uses a 5 ml sample volume.
2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 6A
Internal Standards with Corresponding Assigned Analytes for Quantitation

1st Internal - Fluorobenzene	2nd Internal – Chlorobenzene-d5	3rd Internal – 1,2-Dichlorobenzene-d4
1,1,1-Trichloroethane	1,1,1,2-Tetrachloroethane	1,2,3-Trichlorobenzene
1,1,2-Trichloro-1,2,2-Trifluoroethane	1,1,2,2-Tetrachloroethane	1,2,3-Trichloropropane
1,1-Dicloroethane	1,1,2-Trichloroethane	1,2,4-Trichlorobenzene
1,1-Dichloroethene	1,2-Dibromoethane (EDB)	1,2,4-Trimethylbenzene
1,1-Dichloropropene	1,3-Dichloropropane	1,2-Dibromo-3-chloropropane
1,2-Dichloroethane	2-Hexanone	1,2,-Dichlorobenzene
1,2-Dichloroethene (total)	4-Methyl-2-pentanone (MIBK)	1,3,5-Trimethylbenzene
1,2,-Dichloropropane	Bromoform	1,3-Dichlorobenzene
1,4-Dioxane	Chlorobenzene	1,4-Dichlorobenzene
2,2-Dichloropropane	Chlorodibromomethane	2-Chlorotoluene
2-Butanone (MEK)	Ethyl methacrylate	4-Chlorotoluene
2-Chloroethyl vinyl ether	Ethylbenzene	Bromobenezene
Acetone	Isopropylbenzene	Hexachlorobutadiene
Acetonitrile	m-Xylene & p-Xylene	Naphthalene
Acrolein	o-Xylene	n-Butylbenzene
Acrylonitrile	Styrene	n-Propylbenzene
Allyl chloride	Tetratchloroethene	p-Isopropyltoluene
Benzene	Toluene	sec-Butylbenzene
Bromochloromethane	trans-1,3-Dichloropropene	trans-1,4-Dicloro-2-butene
Bromodichloromethane	Xylenes (total)	tert-Butylbenzene
Bromomethane (Methyl bromide)		
Carbon disulfide		
Carbon tetrachloride		
Chloroethane		
Chloroform		
Chloromethane (Methyl chloride)		
Chloroprene		
cis-1,2-Dichloroethene		
cis-1,3-Dichloropropene		
Cyclohexane		
Dibromomethane (Methylene bromide)		
Dichlorodifluoromethane		
Ethyl ether		
Hexane		
Iodomethane (Methyl iodide)		
Isobutyl alcohol		
Methacrylonitrile		

1st Internal - Fluorobenzene	2nd Internal – Chlorobenzene-d5	3rd Internal – 1,2-Dichlorobenzene-d4
Methyl acetate		
Methyl methacrylate		
Methyl tert-butyl ether (MTBE)		
Methylcyclohexane		
Methylene chloride		
Propionitrile		
tert-Butyl alcohol		
Tetrahydrofuran		
trans-1,2-Dichloroethene		
Trichloroethene		
Trichlorofluoromethane		
Vinyl Acetate		
Vinyl Chloride		

Table 7
Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	25
Dibromofluoromethane	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a standard 5 mL purge Method 8260B, or 10 µg/L for low level Method 8260B waters (which uses a 25 ml sample aliquot), Method 624. For instruments that sample directly from the VOA vial, 10 µL of a 5 µg/mL surrogate solution is added to low level Method 8260B waters, and Method 624 since the instrument uses a 5 ml sample volume.
2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.
3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 8
Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	25
Trichloroethene	25
Toluene	25
Benzene	25
Chlorobenzene	25

Notes:

1) 10 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 standard 5 mL purge Method 8260B water or 10 µg/L for a low level Method 8260B sample when added to a 25 ml sample aliquot.
2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by the QA department.

Table 9
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 10
SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	>0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 11
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

Table 12
Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane (Methyl chloride)	50	52	49
Vinyl chloride	62	64	61
Bromomethane (Methyl bromide)	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane (Methyl iodide)	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Cyclohexane	56	69	84
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 (MTBE)	73	41	
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone (MEK)	43	57	72**
Tetrahydrofuran (THF)	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane (Methylene bromide)	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

Table 12
Characteristic ions

Compound	Primary*	Secondary	Tertiary
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 (MIBK)	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
Isobutanol (Isobutyl alcohol)	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	
Dibromochloromethane	129	208	
1,2,4-Trichlorobenzene	180	182	145

Table 12
Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,1,2-Trichloro-1,2,2-trifluoroethane	101	151	
1,2-Dibromoethane	107	109	
Methyl acetate	43	74	
Methylcyclohexane	83	55	
1,2,3-Trichlorobenzene	180	182	145
1,2,4-Trimethylbenzene	105	120	
1,3,5-Trimethylbenzene	105	120	
1,3-Dichloropropane	76	78	
2,2-Dichloropropane	77	97	
2-Chlorotoluene	126	91	
4-Chlorotoluene	126	91	
Bromobenzene	156	158	77
Bromochloromethane	128	49	130
Hexachlorobutadiene	225	260	227
m-Xylene & p-Xylene	106	91	
Naphthalene	128		
n-Butylbenzene	91	134	
n-Propylbenzene	120	91	
o-Xylene	106	91	
p-Isopropyltoluene	119	134	
sec-Butylbenzene	105	134	
tert-Butylbenzene	119	91	134
Tetrachloroethene	164	129	131

* 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.

Table 13 - 8260B QC Acceptance Criteria															
	Water	LCS			MS			Soil	LCS			MS		
Compound	AMT ug/L	LCL	UCL	RPD	LCL	UCL	RPD	AMT ug/kg	LCL	UCL	RPD	LCL	UCL	RPD
Acetone	40	10	141	32	10	141	32	40	20	150	40	20	150	40
Benzene	40	80	120	20	80	120	20	40	77	120	20	77	120	20
Bromodichloromethane	40	71	119	20	71	119	20	40	70	125	21	70	125	21
Bromoform	40	49	137	20	49	137	20	40	53	140	23	53	140	23
Bromomethane	40	45	150	23	45	150	23	40	25	150	40	25	150	40
2-Butanone	40	31	139	35	31	139	35	40	35	149	36	35	149	36
Carbon disulfide	40	62	126	20	62	126	20	40	50	127	23	50	127	23
Carbon tetrachloride	40	63	139	25	63	139	25	40	69	122	22	69	122	22
Chlorobenzene	40	83	120	20	83	120	20	40	79	120	20	79	120	20
Dibromochloromethane	40	64	124	20	64	124	20	40	70	132	20	70	132	20
Chloroethane	40	33	150	24	33	150	24	40	22	150	40	22	150	40
Chloroform	40	77	119	20	77	119	20	40	72	120	25	72	120	25
Chloromethane	40	49	133	20	49	133	20	40	44	131	27	44	131	27
Cyclohexane	40	69	124	20	69	124	20	40	64	130	21	64	130	21
1,2-Dibromo-3-chloropropane	40	28	150	20	28	150	20	40	35	136	40	35	136	40
1,2-Dibromoethane	40	57	124	20	57	124	20	40	70	131	20	70	131	20
1,2-Dichlorobenzene	40	75	125	20	75	125	20	40	71	124	22	71	124	22
1,3-Dichlorobenzene	40	76	125	21	76	125	21	40	75	118	20	75	118	20
1,4-Dichlorobenzene	40	76	123	20	76	123	20	40	77	116	20	77	116	20
Dichlorodifluoromethane	40	28	140	20	28	140	20	40	25	150	34	25	150	34
1,1-Dichloroethane	40	77	122	22	77	122	22	40	66	124	23	66	124	23
1,2-Dichloroethane	40	63	140	25	63	140	25	40	61	127	23	61	127	23
cis-1,2-Dichloroethene	40	82	116	20	82	116	20	40	80	118	20	80	118	20
trans-1,2-Dichloroethene	40	78	120	20	78	120	20	40	77	121	20	77	121	20
1,1-Dichloroethene	40	69	127	20	69	127	20	40	59	129	25	59	129	25
1,2-Dichloropropane	40	75	114	20	75	114	20	40	72	122	20	72	122	20
cis-1,3-Dichloropropene	40	74	123	20	74	123	20	40	73	120	20	73	120	20
trans-1,3-Dichloropropene	40	63	122	20	63	122	20	40	74	129	20	74	129	20
Ethylbenzene	40	79	124	25	79	124	25	40	78	125	21	78	125	21
2-Hexanone	40	35	129	24	35	129	24	40	32	150	32	32	150	32
Isopropylbenzene	40	73	130	20	73	130	20	40	70	133	22	70	133	22

Table 13 - 8260B QC Acceptance Criteria														
	Water	LCS			MS			Soil	LCS			MS		
Compound	AMT ug/L	LCL	UCL	RPD	LCL	UCL	RPD	AMT ug/kg	LCL	UCL	RPD	LCL	UCL	RPD
Methyl acetate	40	34	127	29	34	127	29	40	27	142	40	10	150	50
Methylcyclohexane	40	67	120	20	67	120	20	40	66	135	23	50	150	50
Methylene chloride	40	75	120	20	75	120	20	40	58	127	28	65	134	20
4-Methyl-2-pentanone	40	33	135	29	33	135	29	40	44	148	30	37	146	39
Methyl tert-butyl ether	40	53	122	20	53	122	20	40	48	132	36	47	131	45
Styrene	40	78	124	22	78	124	22	40	83	129	20	73	121	22
1,1,2,2-Tetrachloroethane	40	59	136	20	59	136	20	40	60	139	24	38	138	20
Tetrachloroethene	40	78	126	25	78	126	25	40	78	129	20	73	120	25
Toluene	40	80	124	20	80	124	20	40	78	124	21	60	134	26
1,2,4-Trichlorobenzene	40	35	150	30	35	150	30	40	51	136	40	48	131	30
1,1,1-Trichloroethane	40	69	134	24	69	134	24	40	67	126	31	71	121	24
1,1,2-Trichloroethane	40	75	126	23	75	126	23	40	70	128	22	61	125	23
Trichloroethene	40	80	120	20	80	120	20	40	76	119	21	52	143	26
Trichlorofluoromethane	40	14	150	20	14	150	20	40	20	150	40	21	153	20
1,1,2-Trichloro-1,2,2-trifluoroethane	40	70	131	30	70	131	30	40	55	130	37	53	146	30
Vinyl chloride	40	57	128	26	57	128	26	40	63	124	27	43	138	25
Xylenes (total)	120	81	121	20	81	121	20	120	83	126	20	75	121	20
4-Bromofluorobenzene	40	75	120		75	120		40	63	120		63	120	
1,2-Dichloroethane-d4	40	62	123		62	123		40	52	124		52	124	
Toluene-d8	40	80	120		80	120		40	72	127		72	127	
Dibromofluoromethane	40	80	120		80	120		40	68	121		68	121	

These limits are established based on internal laboratory data.

19 REQUIREMENTS FOR EPA 624

19.1 Method 624 is required for demonstration of compliance with NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table B-1.

19.1.1 The tune period for this method is defined as 24 hours after passing a 25 ug/ml BFB.

19.1.2 The initial calibration curve for this method requires at least three points.

19.2 Sample concentrations are calculated using the average RRF from the initial calibration curve.

19.2.1 Each target analyte is assigned to the closest eluting internal standard.

19.2.2 Initial demonstration of Proficiency

19.2.3 The spiking level for the four replicate initial demonstration of proficiency is 20 μg/L. The acceptance criteria are listed in Table B-2.

19.3 Initial calibration curve requirements:

19.3.1 Target compounds listed in Method 624 must have RSD ≤ 35%.

19.3.2 If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

19.3.3 For compounds not listed in Method 624, the average response factor will be used for quantitation.

19.3.4 The initial calibration is verified daily by the analysis of a 20 ug/L second source QC Check Standard.

19.4 Continuing calibration verification requirements:

19.4.1 The continuing calibration standard is the daily QC Check Standard. The acceptance criteria are listed in Table B-2. **NOTE: If 8260B and 624 samples are analyzed together the concentration of the CCAL will be 10 ug/L. The**

CCAL will need to pass criteria for both Method 8260B and 624 in order to analyze for both methods using the same CCAL.

19.5 LCS and MS/MSD requirements

19.5.1 The daily 20 ug/L QC Check Standard also serves as the LCS.

19.5.2 The MS and MSD will be 20 ug/L for all compounds.

19.5.3 The recovery limits for MS/MSD and LCS recovery are listed in Table B-2.

19.5.4 The LCS and MS are required for 5% of the samples.

19.6 Method clarifications, modifications and additions

19.6.1 Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

19.6.2 Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.

19.6.3 The source method provides a suggested list of compounds for internal and surrogate standards. TestAmerica Pittsburgh uses the internal standards and surrogates found in Tables 6 and 7.

19.7 When informed that the samples are from a potential chlorinated site, residual chlorine will be checked using total residual chlorine strips. If residual chlorine is detected, the Project Manager will be immediately informed and corrective action will be initiated.

Table A-1.
Method 624 Analytes and Reporting Limits

Analytes	CAS Number	µg/L
Benzene	71-43-2	1
Bromodichloromethane	75-27-4	1
Bromoform	75-25-2	1
Bromomethane	74-83-9	1
Carbon tetrachloride	56-23-5	1
Chlorobenzene	108-90-7	1
Chloroethane	75-00-3	1
2-Chloroethyl vinyl ether *	110-75-8	2
Chloroform	67-66-3	1
Chloromethane	74-87-3	1
Dibromochloromethane	124-48-1	1
1,2-Dichlorobenzene	95-50-1	1
1,3-Dichlorobenzene	541-73-1	1
1,4-Dichlorobenzene	106-46-7	1
1,1-Dichloroethane	75-34-3	1
1,2-Dichloroethane	107-06-2	1
1,1-Dichloroethene	75-35-4	1
trans-1,2-Dichloroethene	156-60-5	1
1,2-Dichloropropane	78-87-5	1
cis-1,3-Dichloropropene	10061-01-5	1
trans-1,3-Dichloropropene	10061-02-6	1
Ethylbenzene	100-41-4	1
Methylene chloride	75-09-2	1
1,1,2,2-Tetrachloroethane	79-34-5	1
Tetrachloroethene	127-18-4	1
Toluene	108-88-3	1
1,1,1-Trichloroethane (1,1,1-Trichloroethene)	71-55-6	1
1,1,2-Trichloroethane (1,1,2-Trichloroethene)	79-00-5	1
Trichloroethene (Trichloroethane)	79-01-6	1
Trichlorofluoromethane	75-69-4	1
Vinyl chloride	75-01-4	1

***** 2-Chloroethylvinyl ether degrades under acidic conditions and cannot be determined in an acid preserved sample.

Table A-2.
Method 624 QC Acceptance Criteria

Analytes	Daily QC Check acceptance criteria %Recovery	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike acceptance criteria (% Recovery)
Benzene	64-136	15.2-26.0	6.9	37-151
Bromodichloromethane	65-135	10.1-28.0	6.4	35-155
Bromoform	71-129	11.4-31.1	5.4	45-169
Bromomethane	14-186	D-41.2	17.9	D-242
Carbon tetrachloride	73-127	17.2-23.5	5.2	70-140
Chlorobenzene	66-134	16.4-27.4	6.3	37-160
Chloroethane	38-162	8.4-40.4	11.4	14-230
2-Chloroethyl vinyl ether	0-224	D-50.4	25.9	D-305
Chloroform	67-133	13.7-24.2	6.1	51-138
Chloromethane	D-204	D-45.9	19.8	D-273
Dibromochloromethane	67-133	13.8-26.6	6.1	53-149
1,2-Dichlorobenzene	63-137	11.8-34.7	7.1	18-190
1,3-Dichlorobenzene	73-127	17.0-28.8	5.5	59-156
1,4-Dichlorobenzene	63-137	11.8-34.7	7.1	18-190
1,1-Dichloroethane	72-128	14.2-28.5	5.1	59-155
1,2-Dichloroethane	68-132	14.3-27.4	6.0	49-155
1,1-Dichloroethene	50-150	3.7-42.3	9.1	D-234
trans-1,2-Dichloroethene	69-131	13.6-28.5	5.7	54-156
1,2-Dichloropropane	34-166	3.8-36.2	13.8	D-210
cis-1,3-Dichloropropene	24-176	1.0-39.0	15.8	D-227
trans-1,3-Dichloropropene	50-150	7.6-32.4	10.4	17-183
Ethylbenzene	59-141	17.4-26.7	7.5	37-162
Methylene chloride	60-140	D-41.0	7.4	D-221
1,1,2,2-Tetrachloroethane	60-140	13.5-27.2	7.4	46-157
Tetrachloroethene	73-127	17.0-26.6	5.0	64-148
Toluene	74-126	16.6-26.7	4.8	47-150
1,1,1-Trichloroethane (1,11-Trichloroehene)	75-125	13.7-30.1	4.6	52-162
1,1,2-Trichloroethane (1,1,2-Trichloroethene)	71-129	14.3-27.1	5.5	52-150
Trichloroethene (Trichloroethane)	66-134	18.6-27.6	6.6	71-157
Trichlorofluoromethane	48-152	8.9-31.5	10.0	17-181

Table A-2.
Method 624 QC Acceptance Criteria

Analytes	Daily QC Check acceptance criteria %Recovery	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike acceptance criteria (% Recovery)
Vinyl chloride	4-196	D-43.5	20.0	D-251

D = MDL for the particular analyte

Note: These limits are based on method 624. The QC check acceptance criteria in percent recovery is calculated from the concentration range given in the method where the QC sample concentration is at 20 ug/L. For instance for Benzene the method states a concentration range of 12.8-27.2 ug/L. 12.8/20 *100 = 64 and 27.2/20 * 100 = 136, therefore these conversions in percent recovery is listed in the above table.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION

METHODS: SW846 7470A AND MCAWW 245.1

Approvals (Signature/Date):

Larry Matko	09/07/10 Date	Steve Jackson	09/01/10 Date
Technical Manager		Health & Safety Manager / Coordinator	
Nasreen DeRubeis	08/31/10 Date	Albert F. Vicinie	08/31/10 Date
Quality Assurance Manager		Laboratory Director	

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. Both the water bath digestion and the hot block digestion are available at the TestAmerica Pittsburgh facility, however the default practice is the hot block digestion for 7470A. The water bath procedure is always used for 245.1. Both procedures are described in this SOP.

1.2. 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.

1.3. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed, however Method 7471A/B (see PT-MT-007) is usually the method of choice. All matrices require sample preparation prior to analysis.

1.4. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.5. The TestAmerica reporting limit for mercury in aqueous matrices is 0.0002 mg/L.

1.6. For DoD QSM Version 3 requirements, refer to SOP PT-QA-025 and for DoD QSM Version 4.1 requirements, refer to SOP PT-QA-029.

1.7. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.1 in the Quality Assurance Manual.

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.

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. Seawaters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation 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 head space 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 Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2. 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. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

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
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn 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.
Stannous Chloride	Irritant	2 Mg/M3 TWA as Tin	Causes irritation to the respiratory tract. Can irritate skin and eyes. Symptoms include coughing and shortness of breath. Contact with skin and/or eyes may cause redness, itching and pain.
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.			

5.5. 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.

5.6. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.6.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.6.2. Iodine, 0.25%, in a 3% KI solution.

5.7. 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.

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. 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 or EH&S coordinator.

5.10. 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.

5.11. 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) or hot block capable of maintaining a temperature of 95 ± 5°C for 2 hours.

6.2. Leeman HYDRA AA Automated Mercury Analysis System.

6.3. Disposable Sealable Sample Containers (Corning).

6.4. Argon gas supply (ultrahigh purity-grade).

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 (1000 ppm) mercury standards (in 10% HNO_3) are purchased as custom TestAmerica 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. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be made monthly and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) 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. A second source working standard is prepared at 0.1 ppm for preparation of the ICV.

7.5. The calibration standards listed in Table I must be prepared fresh daily from the working standard (7.4) 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. The 0, .5, 1.0, 5.0 and 10 standards are recommended by Thermo Electron. The 0.2 standard level was selected to include a standard at the RL. See Table 1 (Appendix A) for the preparation of the ICV, CCV and RLV standards.

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.

7.6. The initial calibration verification standard (ICV) must be made from a different stock solution than that of the calibration standards.

7.7. 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.

7.8. 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.

7.9. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.10. Stannous chloride solution: Add 200 g of stannous chloride to 2 L of 10% hydrochloric acid.

7.11. Stannous sulfate may be used in place of stannous chloride. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

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.

7.14. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis. For TCLP leachates, the holding time is 28 days from the time of TCLP extraction 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.

8.3. Dissolved metals samples that are filtered and preserved at the laboratory with concentrated Nitric acid will be held for 24 hours before digestion.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 12.0.

9.2. Preparation Batch - A group of up to 20 samples composed of the same matrix and 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 for 7470A or a matrix spike (one per 10 or fewer samples) for 245.1. 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 limit, or above 10% of either the measured concentration of that analyte in associated samples or the regulatory limit. See SOP PT-QA-021 for more detail on criteria and corrective actions. In addition, blank contamination should always be evaluated against project specific requirements. **Refer to PT-QA-025 (QSM 3.0) or PT-QA-029 (QSM 4.1) for specific DoD requirements for the method blank.**

- 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 taken in consultation with the client and 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 and the client must be notified.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS (referred to as a Laboratory Fortified Blank in 245.1) 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. The CCV results can be reported as LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits the system is out of control and corrective action must occur. Corrective action will result in the batch being re-prepped and re-analyzed. In-house control limits are 80 - 120% for SW-846 method 7470A and 85 – 115% for EPA method 245.1.

- In the instance where the LCS recovery is > 120% (7470A) or > 115% (245.1) and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case 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 of up to 20 samples for 7470A or a MS must be processed for every 10 or fewer samples for 245.1. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added (referred to as a Laboratory Fortified Matrix in 245.1). 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 % (7470A) or 70 – 130% (245.1) recovery and 20% RPD must be applied to the MS/MSD. **Refer to PT-QA-025 (QSM 3.0) or PT-QA-029 (QSM 4.1) for specific DoD requirements for 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% (7470A) or 5% (245.1) 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. **Refer to PT-QA-025 (QSM 3.0) or PT-QA-029 (QSM 4.1) for specific DoD requirements for the ICB.** If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the associated samples. The ICV is equivalent to the Quality Control Sample (QCS) and the first Initial Performance Check (IPC) specified in 245.1.

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 and at the end of the analytical sequence. The CCV must be a mid-range

standard at a concentration other than that of the ICV. **The CCV result must fall within 10% (7470A and 245.1) of the true value for that solution.** If both methods are analyzed in the same sequence the tighter criteria of 10% is used for the CCV. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. **Refer to PT-QA-025 (QSM 3.0) or PT-QA-029 (QSM 4.1) for specific DoD requirements for the CCB.** Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.9. Reporting Limit Verification Standard (RLV) – Calibration accuracy at the laboratory reporting limit is verified after the analysis of the ICB. Until in-house control limits are established, a control limit of 50 – 150% recovery will be applied.

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 10.3.11 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. PROCEDURE

10.1. Calibration and Standardization

10.1.1. Calibration standards must be processed through the preparation procedure as described in Section 10.2.

10.1.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.1.3. 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.

10.1.4. 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 CVAA instrument manual for detailed setup and operation protocols.

10.1.5. 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 reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.1.6. The calibration curve must have a correlation coefficient of $\geq$0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

10.1.7. Refer to Section 9.0 and Table II for calibration verification procedures, acceptance criteria and corrective actions. The NELAC requirement for verification of the initial calibration at varied concentrations is met daily since the ICVs, CCVs, and RLVs are all at different concentrations.

10.2. Sample Preparation:

10.2.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB, RLV) are processed through the digestion procedure as well as the field samples. *An exception to this is for Method 245.1 samples. The calibration curve samples are* ***not*** *heated.*

10.2.2. Transfer 50 mL of well-mixed sample or standard to a clean sample digestion bottle. Refer to PT-QA-024 for subsampling procedures.

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.

10.2.3. Add 2.5 mL of concentrated H_2SO_4 and 1.25 mL of concentrated HNO_3 mixing after each addition.

Note: All spiking (LCS, MS, MSD) should be done after the initial addition of acids (see Appendix A, Table 1).

10.2.4. Add 7.5 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 15 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 associated samples, standards, *and QC samples (e.g. LCS and blank)* 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.

10.2.5. Add 4 mL of potassium persulfate solution for a 50 mL sample and heat for two hours in a water bath at 90 - 95 °C. (**Note:** 8 mL of potassium persulfate solution would be used for a 100 mL sample, etc. for proportional volumes).

NOTE: Alternatively, for analyses using 7470A, samples may be digested using a hot block capable of maintaining a temperature of 95 $\pm$ 5°C for 2 hours.

10.2.6. Cool samples.

10.3. Sample Analysis:

10.3.1. Refer to the SOP PT-MT-010 and the instrument manuals for detailed setup and operation protocols for the LEEMAN Hydra AA.

10.3.2. Refer to CVAA instrument manual for detailed setup and operation protocols.

10.3.3. When ready to begin analysis, add 6 mL of sodium chloride-hydroxylamine hydrochloride “clearing solution” to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Add this solution in 6 mL increments until the permanganate is completely reduced i.e. colorless.

10.3.4. Automated determination: Follow instructions provided by instrument manufacturer.

10.3.5. 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.

10.3.6. All measurements must fall within the defined calibration range to be valid. When sample concentrations exceed the upper limit of the calibration curve, the samples will be diluted and reanalyzed (if possible) to bring them within calibration curve. When reported sample concentrations either exceed the upper limit of the curve (i.e. cannot be rerun) or fall below the reporting limit, the data will be qualified as estimated. 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.

10.3.7. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.3.8. 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.

10.3.9. The following analytical sequence must be used with 7470A and 245.1:

Instrument Calibration

ICV

ICB

RLV

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 Methods 7470A and 245.1.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

10.3.10. The following run sequence is consistent with 7470A, CLP and 245.1 and may be used as an alternate to the sequence in 10.3.9. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration

ICV

ICB

RLV or 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 the appropriate CLP SOPs (PT-MT-006) for quality control requirements for QC samples.

* Refer to the CLP SOPs for information on the CRA.

10.3.11. 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 E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

10.4. 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.

10.5. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and parts maintenance. For instrument troubleshooting, use the auto diagnostics software. If the problem cannot be determined using the software, place a call to service personnel.

10.6. 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.

10.7. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11. DATA ANALYSIS AND CALCULATIONS

11.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

11.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

11.3. RLV percent recoveries are calculated using the same equation as the ICV or CCV (replace ICV or CCV with RLV in the above equations).

11.4. 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

11.5. 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

11.6. 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

11.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

11.8. Appropriate factors must be applied to sample values if dilutions are performed.

11.9. Sample results should be reported with up to three significant figures in accordance with the TestAmerica significant figure policy.

12. METHOD PERFORMANCE

12.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7470A or the 245.1, the following requirements must be met.

12.1.1. 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 determined in accordance with 40 CFR Part 136 Appendix B requirements and SOP PT-MT-007. The result of the MDL must be below the TestAmerica reporting limit.

12.1.2. Initial Demonstration Study - This requires the analysis of four LCS samples. The LCS 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. Refer to SOP PT-QA-001.

12.1.2.1. Four aliquots of the LCS are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

12.2. 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 % (7470A) or +/- 30% (245.1) and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.4. **Refer to PT-QA-025 (QSM 3.0) or PT-QA-029 (QSM 4.1) for specific DoD requirements for the method blank and MS.** The laboratory control sample should

recover within 20% (7470A) or 15% (245.1) of the true value until in house limits are established.

12.3. Training Qualification:

12.3.1. 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.

13. POLLUTION PREVENTION

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

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 PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1. Extracted sample containing less than 1 ppb Hg. This waste is collected in waste containers identified as "Acid Waste", Waste #33. It is neutralized to a pH between 6 and 9 and is disposed down a lab sink.

14.1.2. Unused Standards. This waste collected in containers identified as "Mercury Standards Waste", Waste #4.

14.1.3. Extracted sample containing greater than 1 ppb Hg. This waste collected in containers identified as “Mercury Standards Waste”, Waste #4.

14.1.4. Mercury Analyzer Waste. Waste discharged from mercury analyzer is collected in containers identified as “Mercury Standards Waste”, Waste #4.

15. REFERENCES/CROSS-REFERENCES

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

15.2. “Methods for the Chemical Analysis of Water and Wastes”, EPA-600/4-79-020, U.S.EPA, 1994, Method 245.1, Revision 3.0.

15.3. U.S.EPA Statement of Work for Inorganics Analysis, ILM04.1.

15.4. PT-QA-001, Employee Orientation and Training.

15.5. PT-QA-006, Procurement of Standards and Materials; Labeling and Traceability.

15.6. PT-QA-007, Method Detection Limits.

15.7. PT-QA-009, Rounding and Significant Figures.

15.8. PT-QA-016, Nonconformance & Corrective Action System.

15.9. PT-QA-018, Technical Data Review Requirements.

15.10. PT-QA-021, Quality Assurance Program.

15.11. PT-QA-022, Equipment Maintenance

15.12. PT-QA-024, Subsampling.

15.13. PT-QA-025, DoD QSM Version 3.

15.14. PT-QA-027, Sample Receiving and Chain of Custody.

15.15. PT-QA-029, QA/QC Requirements for DoD QSM Version 4.1.

15.16. PT-MT-010, Operation of Leeman PS200 (Automated) for Mercury Analysis.

15.17. PT-LQAM- Pittsburgh Laboratory Quality Assurance Manual.

16. **METHOD MODIFICATIONS:**

16.1. Modifications/Interpretations from reference method.

16.1.1. Modifications from both 7470A and 245.1.

16.1.1.1. 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.

16.1.1.2. 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.”

16.1.1.3. The alternate run sequence presented in Section 10.3.10 is consistent with method requirements.

16.1.2. Modifications from Method 7470A

16.1.2.1. 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. **Refer to PT-QA-025 (QSM 3.0) or PT-QA-029 (QSM 4.1) for specific DoD requirements for the method blank.**

16.1.2.2. Documentation is on file from EPA’s Office of Solid Waste (Oliver Fordham 11/28/95) regarding the acceptance of the autoclave as an equivalent heating device to the water bath. In his letter, Mr. Fordham cited the CLP water protocol 245.1 CLP-M and therefore the operating parameters from that method were adopted for 7470A (15 minutes at 120 °C and 15 lbs.).

16.1.3. Modifications from 245.1

16.1.3.1. Method 245.1, Section 9.3 states concentrations should be reported as follows: Between 1 and 10 ug/L, one decimal; above 10 ug/L, to the nearest whole number. TestAmerica reports all Hg results under this SOP to two significant figures.

17. ATTACHMENTS

17.1. 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.
- Non-conformance summary (if applicable).

17.2. APPENDIX A - TABLES

17.3. APPENDIX B - TestAmerica Hg DATA REVIEW CHECKLIST

17.4. APPENDIX C - MSA GUIDANCE

17.5. APPENDIX D – PARTS MAINTENANCE GUIDE

17.6. APPENDIX E- CONTAMINATION CONTROL GUIDELINES

17.7. APPENDIX F - PREVENTIVE MAINTENANCE

18. REVISION HISTORY

18.1. Revision 8, 9/2/2008

18.1.1. Updated the Headers to the new Corporate format; updated SOP and section references throughout the SOP; added Corporate text to the Scope, Safety, Pollution Control and Waste Management sections; changed STL to TestAmerica throughout the SOP; updated the Reference section.

18.1.2. Addedto Section 8.3: Dissolved metals samples that are filtered and preserved at the laboratory with concentrated Nitric acid will be held for 24 hours before digestion.

18.2. Revision 9, 10/8/2009

18.2.1. Removed all reference to the autoclave and add the reference for the hot block digestion requirements throughout the SOP.

18.2.2. Added the SOP reference for DoD QSM 4.1, PT-QA-029 in the appropriate areas within the SOP. Added reference to SOP PT-QA-022, Equipment Maintenance.

18.2.3 In section 10.2.2 changed 100 mL to 50 mL.

18.2.4 In section 10.2.3 changed 5 mL to 2.5 mL and 2.5 mL to 1.25 mL.

18.2.5 In section 10.2.4 changed 15 mL to 7.5 mL.

18.2.6 In section 10.2.5 changed 8 mL to 4 mL.

18.2.7 Updated Figure 1/Figure 2 to remove the autoclave requirements and add the hot block requirements.

18.2.8 Updated Table 1 to separate out the Working Standard requirements for 7470A and 245.1.

18.3. Revision 10

18.3.1. Updated the SOP reference for 7471A/B to PT-MT-007 in section 1.3.

18.3.2. Removed reference to the LEEMAN PS200II Mercury Analyzer in sections 6.2 and 10.3.1 since this instrument has been taken out of service.

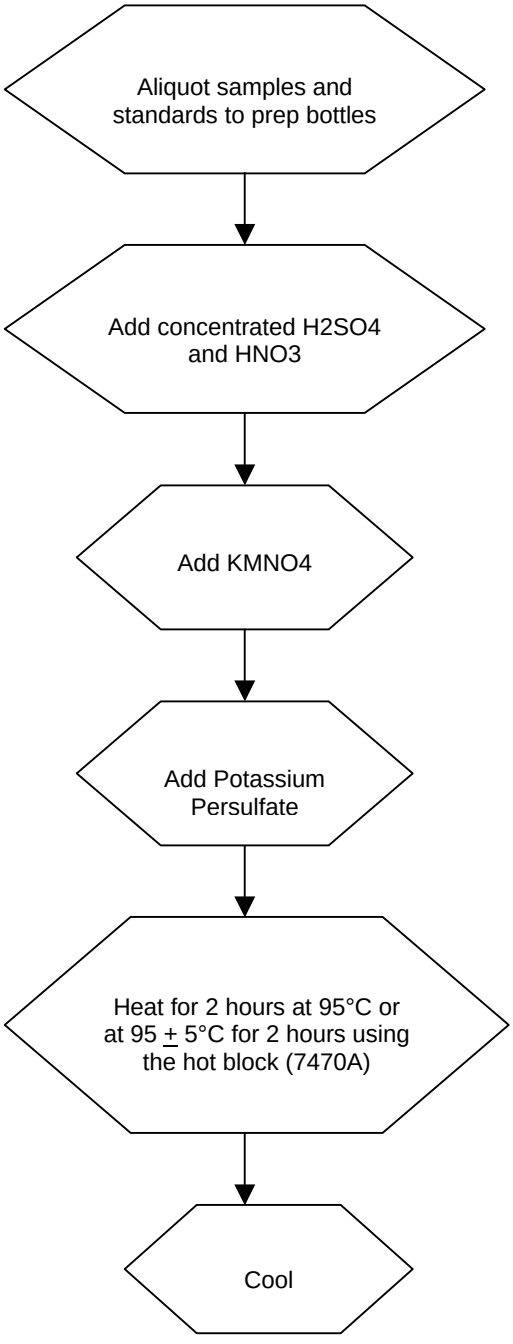
18.3.3. Added to section 9.8: If both methods are analyzed in the same sequence the tighter criteria of 10% is used for the CCV.

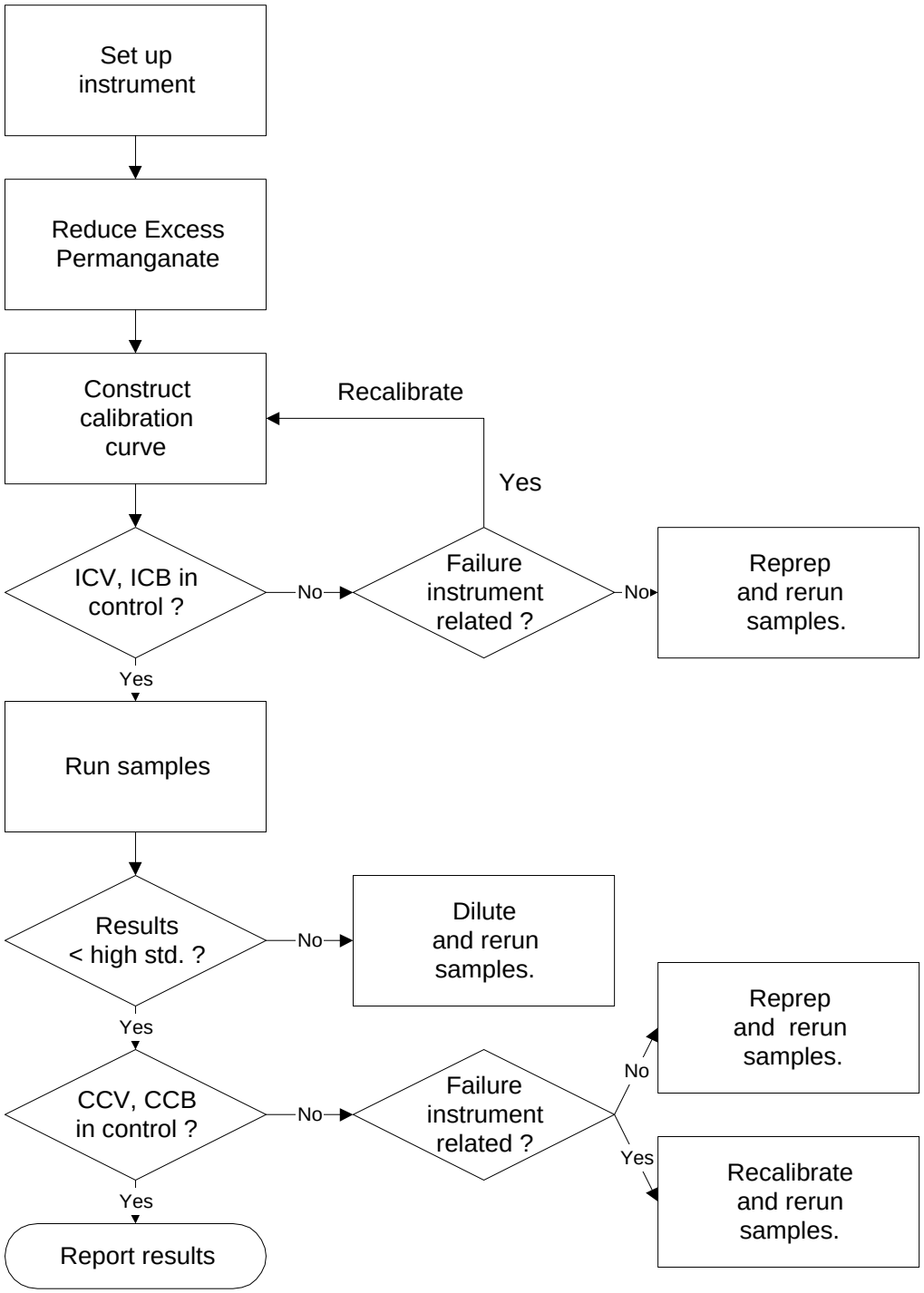
18.3.4. In section 10.2.5, corrected the potassium persulfate portion to 4 mL for a 50 mL sample.

18.4. Revision 11

18.4.1. Updated CCV criteria to ±10% for 7470A in section 9.8 and Table II.

Figure 1. Aqueous Sample Preparation – Mercury / **Figure 2**. CVAA Mercury Analysis




Set up instrument
Reduce Excess Permanganate
Construct calibration curve
Recalibrate
Yes
ICV, ICB in control ?
No
Failure instrument related ?
No
Reprep and rerun samples.
Yes
Run samples
Results < high std. ?
No
Dilute and rerun samples.
Yes
CCV, CCB in control ?
No
Failure instrument related ?
No
Reprep and rerun samples.
Yes
Recalibrate and rerun samples.
Yes
Report results

APPENDIX A

TABLES

TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (MG/L)

Method		Reporting Limit	
SW846 7470A		0.0002 mg/ L	
SW846 7470A (TCLP)		0.0002 mg/ L	
MCAWW 245.1		0.0002 mg/ L	
Standard or QC sample	**7470A mLs of 0.1 ppm Working Standard**	**245.1 mLs of 0.1 ppm Working Standard**	**Concentration (mg/L)*** **
Std 0	0	0	0
Std 1	0.1	0.2	0.0002
Std 2	0.25	0.5	0.0005
Std 3	0.5	1.0	0.001
Std 4	2.5	5.0	0.005
Std 5	5.0	10.0	0.010
ICV	1.25	2.5**	0.0025
CCV	2.5	5.0	0.005
RLV	0.1	0.2	0.0002
LCS	1.25	2.5	0.0025
Aqueous MS	0.5	1.0	0.001
TCLP MS	0.25	5.0	0.005

* SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements.

** Prepared from a second source 0.1 ppm working standard.

*** When brought to a 100 mL final volume.

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	7470A: 90 - 110 %. 245.1: 95 – 105%	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
RLV	Beginning of every analytical run, immediately following the ICB.	50 – 150% recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.9).
CCV	Every 10 samples and at the end of the run.	7470A: 90 - 110%. 245.1: 90 – 110%	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (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. (1) 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.

TABLE II. Summary Of Quality Control Requirements

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% (7470A) or 85 – 115% (245.1) 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 (7470A) or one for every 10 or fewer samples (245.1).	75 - 125 % (7470A) or 70 – 130% (245.1) recovery or in-house control limits. [(1)] 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 10.3.11
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % (7470A) or 70 – 130% (245.1) recovery or in-house control limits; RPD ≤ 20%. [(1)] (See MS)	See Corrective Action for Matrix Spike.

[(1)] For specific DoD requirements, refer to PT-QA-025 (QSM 3.0) or PT-QA-029 (QSM 4.1).

APPENDIX B
Example Hg DATA REVIEW CHECKLIST

Run Date: _____ Lots Analyzed: 4. _____ 8. _____ 12. _____

Analyst: _____ 1. _____ 5. _____ 9. _____ 13. _____

Instrument: _____ 2. _____ 6. _____ 10. _____ 14. _____

Methods: _____ 3. _____ 7. _____ 11. _____ 15. _____

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)	Comments
A. Calibration/Instrument Run QC					
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?					

APPENDIX B - DATA REVIEW CHECKLIST

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Level Review (✓)	Comments
5. Date/Time of analysis verified as correct?					

General

Comments:_____
Analyst & Date: _____ Second-Level Review & Date: _____

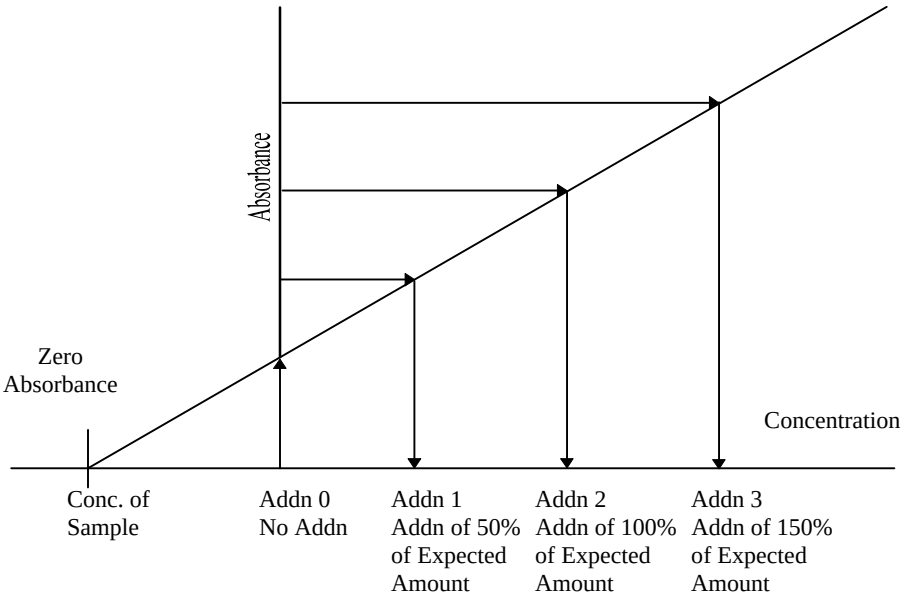
APPENDIX C. MSA GUIDANCE

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. PARTS MAINTENANCE GUIDE

Maintenance Schedule

The software offers a simple to use online Scheduled Maintenance page. To view the page go to Instrument:Scheduled Maintenance (F1 Menu, I, S). A page displaying all items necessary to keep the instrument well maintained is shown (see figure 6.1A).

```
RunProt:
RunFold:          Seq: 0    Batch:
                  Prnt: R/T Off
                  Rev: 3.390  15:40:47 14 Jan 1996 Xmit: Off  Gas:        LPM
 None                                              User:              A/S: On
INSTRUMENT:  Scheduled Maintenance
                                    Uses left     Last service    Next service
 replace: Pump tubing                   200       14-Jan-96       24-Jan-96
          Waste drain tubing           2500       14-Jan-96       29-Dec-96
          Liquid/gas separator         5000       14-Jan-96       14-Mar-96
          puMp head                   10000           N/A             N/A
          Hg lamp                       N/A       14-Jan-96       12-Jun-96
          Reductant bottle              400       14-Jan-96       12-Jul-96
          prOcess tubing               5000           N/A             N/A
 Clean optical cell                     300           N/A             N/A
 clean External optics                  N/A       14-Jan-96       12-Jul-96


* - needs maintenance

For help on <hotkey> press Shift <hotkey>
```

Figure 6.1a. Scheduled maintenance screen

Each scheduled maintenance item has a usage counter, timed usage, or both (N/A indicates that the usage counter or the timed usage is not applicable for that item). If either condition expires for a given item a maintenance message will alert the user at the top of the status box.

Maintenance Procedures

An asterisk(*) will appear next to the item requiring maintenance on the Scheduled Maintenance screen. To clear the message hit <Tab> or replace, clean, or replenish the item using the hot key for the item on the Scheduled Maintenance page. To perform the maintenance on a given item simply type the hot key (e.g. Type <P> for Pump tubing) and follow the directions. Once the directions are followed to completion, the usage counter and timed usage gets updated.

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 20% 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

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 (Hydra AA)

Daily	As Needed	Annually
Clean lens windows with methanol.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture reading.	Check pump tubing/drain tubing.	Check liquid/gas separator.
Check argon flow/pressure.	Clean optical cell	
Check tubing and replace, if needed.	Lubricate pump	
Check drain.		
Replace drying tube.		

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) AND SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLLP)

METHODS: SW-846 1311 AND 1312

Approvals (Signature/Date):

Brian A. Pino 03/11/09	01/21/09
Brain Pino Date	Steve Jackson Date
Technical Manager	Health & Safety Manager / Coordinator
Nasreen K. DeRubeis 01/19/09	03/11/09
Nasreen DeRubeis Date	Albert F. Vicinie for Larry Matko Date
Quality Assurance Manager	General Manager/Laboratory Director

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.

1.2.1 **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) that 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. Any deviations from the method affecting these parameters must be documented as a nonconformance, with a cause and corrective action described.

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 that 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.

1.10 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.1 in the Quality Assurance Manual.

1.11 For subsampling procedures refer to SOP PT-QA-024.

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. 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 phase 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 TCLP solution generated from this procedure.

3.2 "Percent Wet Solids" is that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

4. INTERFERENCES

4.1 Oily wastes may present unusual filtration and drying problems. As recommended by EPA (see Figure 3), oily wastes will 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.

4.2 Wastes containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the oil exceeds TCLP 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 the Section 9.0 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 Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 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
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.
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.
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 become contaminated will be removed and discarded, other gloves will be cleaned immediately.

5.4 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.

5.5 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.

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 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.

5.9 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.

5.10 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 or EH&S coordinator.

5.11 Due to the potential for ignition and/or flammability, do not attempt to dry non-aqueous liquid samples in an oven.

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 plastic-coated, with Teflon lid inserts) or 2 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, capable of 0 - 50 psi 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, 0.7 μm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use. As an alternative, certified pre-washed filters may be used. 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 Devices are used to collect the initial liquid phase and the final extract of the waste from the ZHE device, either of the following may be used:

6.5.1 Gas-tight syringes, 100 mL capacity, Hamilton 0158330 or equivalent, or

6.5.2 Tedlar bags

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 pH probes.

6.9 Magnetic stirrer/hotplate and stirring bars.

6.10 VOA vials, 40 mL, with caps and septa.

6.11 Glass jars, 1/2 - 1 gallon, with Teflon lid-inserts.

6.12 Nalgene plastic bottles, 1 liter.

6.13 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 83.3 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 Nitric acid, concentrated, reagent grade liquid (HNO_3).

7.4 Sodium hydroxide, 1 N, purchased or prepared: 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.

7.5 Acetic acid, glacial: concentrated, reagent grade liquid (HOAc).

7.6 pH calibration solutions: buffered to a pH of 4, 7, and 10. Commercially available. Fresh buffer solution must be used each day of analysis.

7.7 TCLP Leaching Fluids

7.7.1 General Comments

7.7.1.1 The pH of both solutions listed below should be monitored daily and the pH probes are to be calibrated prior to use.

7.7.1.2 TCLP Leaching Fluids are purchased from Ricca Chemical Company or prepared following the procedure outlined below.

7.7.1.3 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.

7.7.1.4 Additional volumes of extraction fluids listed above may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.

7.7.2 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. When correctly prepared, the pH of this solution is 4.93 ± 0.05.

7.7.3 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. When correctly prepared, the pH of this solution is 2.88 ± 0.05.

7.8 Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.

7.9 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.

7.10 SPLP Leaching fluids

7.10.1 SPLP Leaching Fluids are purchased from Lab Chem Inc. or prepared following the procedure outlined below. 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.

7.10.2 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 wastewaters.

7.10.3 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.

7.10.4 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.

7.11 Methanol and methylene chloride - used to aid in cleaning oil-contaminated equipment.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT 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.

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 ^{o}C). Samples should be opened only immediately prior to extraction.

8.4 Samples should be refrigerated to 4 ± 2 ^{o}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 Batching Samples - Groups of samples with visibly different bulk matrices (e.g., petroleum sludge and soil samples) must be batched separately for QC testing purposes.

9.3 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). Extraction vessels will be uniquely numbered. Each time a new batch is set up the blank should be rotated sequentially to the next vessel to ensure all vessels are periodically checked. Consult the TestAmerica QC Program and the individual analysis SOPs for blank acceptance criteria.

9.4 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. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).

9.5 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.

9.5.1 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.

9.5.2 The use of internal calibration or alternate methods may be needed when the recovery of the matrix spike is below the expected performance (see Section 9.6.2).

9.5.3 Consult the individual analysis SOPs for additional guidance on spike compounds and levels.

9.6 Corrective Actions

9.6.1 Consult the TestAmerica QC Program and individual analysis SOPs for corrective action for blanks and LCS

9.6.2 Method of Standard Additions (MSA) shall be used for metals if all of the following conditions are met:

9.6.2.1 Recovery of the analyte in matrix spike is not at least 50%,

9.6.2.2 The concentration of the analyte does not exceed the regulatory level, and

9.6.2.3 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.

9.7 Refer to PT-QA-025 for specific DoD QC requirements**.**

10. PROCEDURE

10.1 GENERAL COMMENTS

10.1.1 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 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.

10.1.2 Refer to SOP PT-QA-024 for subsampling procedures.

10.2 PRELIMINARY SAMPLE EVALUATIONS (Refer to Flow Chart #1, Appendix D)

10.2.1 Preliminary TCLP evaluations (percent solids, particle size, selection of extraction fluid, and fluid/leachate compatibility) are required to be done using a minimum of a 100 gram aliquot of waste. This aliquot may also undergo the actual TCLP or SPLP extraction for Non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.

10.2.2 Consult the holding times for the appropriate tests (Section 8.7) and prioritize extractions such that holding times are not exceeded.

10.2.3 Determine the total volume of TCLP leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to the following:

Table 2. Minimum Required Leachate Volume

Analysis	Required Volume (mL)
Volatiles	2 x 40
Semi-volatiles	200
Pesticides	100
Herbicides	100
Metals	150

10.2.3.1 Determine the total volume of TCLP leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to the for SPLP - similar volumes are required for volatiles and metals. If semivolatiles, pesticides or herbicides are required, a full 1 L volume must be prepared for each test requested.

10.2.3.2 For TCLP and SPLP samples used for matrix spike and matrix spike duplicate analysis, three times the listed volumes are required.

10.2.4 SAMPLE DESCRIPTION

10.2.4.1 Observe the number of phases present in the sample according to apparent density. (Note: It may be impossible to distinguish apparent density if only one liquid phase is observed and there is no indication on the COC form. If this is the case, record it as aqueous material and let the subsequent analytical record show if the liquid is organic.) It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase.

10.2.4.2 If the sample has multiple phases and is received in more than 1 bottle then the contents of each bottle should be combined in a single larger container prior to processing the sample further. If this is not possible, then the alternate procedure described in the following section should be used.

10.2.4.3 Properly record the relative amounts of each phase by measuring the depth of the layers in each container after the contents have been allowed to settle. Determine the combined volume of each phase for all containers. These values are needed to determine the correct volume/mass adjustments on the final result. This procedure is not appropriate if testing will be done for volatile organic compounds.

10.2.5 PERCENT SOLID PHASE

10.2.5.1 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 10.4 (Procedure: ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.

10.2.5.2 Determine Type of Filtration Apparatus Needed

10.2.5.2.1 If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 10.2.6 (Particle-size Reduction).

10.2.5.2.2 If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a highly granular, liquid containing waste vacuum filtration may be used.

10.2.5.2.3 If the sample is viscous (sludge or has high solids content), use pressure filtration.

10.2.5.3 Weight of filter - Measure and record this value before loading the filter into the filter holder.

10.2.5.4 Weight of subsample and filtrate for percent solids measurement.

10.2.5.4.1 Assemble the filtration apparatus (use blunt forceps to handle the 0.6 to 0.8 μm filter membrane).

10.2.5.4.2 Homogenize the waste in the sample container. Measure and record the gross weight.

10.2.5.4.3 Measure and record the tare weight of the filtration vessel.

10.2.5.4.4 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 sample container and any residual sample.

10.2.5.4.5 Calculate and record the net weight of sample used for testing.

10.2.5.5 Filtration for percent solids

10.2.5.5.1 Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter during a 2 minute period.

10.2.5.5.2 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 2 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 does not pass through the filter is classified as a solid

10.2.5.5.3 Remove the filtrate collection vessel, weigh and record the gross weight.

10.2.5.5.4 Calculate and record the net weight of filtrate. This result will be used in the percent solids calculation.

10.2.5.5.5 Pour the filtrate into a graduated cylinder. Measure and record the volume of the aqueous phase. Measure and record the volume of any organic phase. If more than one organic phase is observed, provide a description. These results will be used in the final sample calculations.

10.2.5.5.6 Retain the filtrate for use in Section 10.2.8 (Determination of Filtrate/Extraction Fluid Compatibility), and for possible recombination with the filtrate obtained in Section 10.3.

10.2.5.6 Percent of Wet Solids

10.2.5.6.1 Subtract the net weight of the filtrate from the net weight of the subsample to calculate the total weight of wet solids.

10.2.5.6.2 Calculate the weight percent of wet solids using the Equation in Section 11.1.1.

10.2.5.6.3 If the percent wet solids result is $\geq$ 0.5% and < 5.0%, and it is noticed that a small amount of the aqueous filtrate is entrained in the wetting of the filter, proceed to Section 10.2.5.7 to complete the percent solids measurement on a dry-weight basis. **Note:** If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter; proceed to Section 10.2.6 (Particle-Size Reduction).

10.2.5.6.4 If the percent wet solids result is greater than 5.0%, proceed to Section 10.2.6 (Particle-Size Reduction).

10.2.5.6.5 If the percent wet solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is equivalent to the final leachate.

10.2.5.7 Weight percent of dry solids (skip this step and refer to Appendix B for oily samples). **Note**: These steps are required only if it is noticed that a small amount of the filtrate is entrained in wetting of the filter and the percent wet solids content is ≥ 0.5% and < 5.0 %

10.2.5.7.1 Remove the filter with the wet solids from the filtration apparatus.

10.2.5.7.2 Dry the filter and solid phase at 100 ± 20 ° C.

10.2.5.7.3 Remove the filter from the oven and allow to cool in a desiccator.

10.2.5.7.4 Weigh and record the gross dry weight.

10.2.5.7.5 Repeat the drying step. Weigh and record the second gross dry weight. If the two weighings do not agree within 1%, perform additional drying and weighing until successive weighings agree within 1%.

10.2.5.7.6 Calculate the weight percent of dry solids.

10.2.5.7.7 If the dry solids result is ≥ 0.5% and the sample will be extracted for non-volatile constituents, proceed to Section 10.2.6 (Particle Size Reduction) using a fresh wet portion of waste.

10.2.5.7.8 If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Proceed to Section 10.2.8 (Determination of Filtrate/Leachate Compatibility) to determine whether or not the material is a non-aqueous, immiscible liquid.

10.2.6 PARTICLE-SIZE REDUCTION FOR FLUID SELECTION

10.2.6.1 The subsample used for fluid selection must consist of particles less than 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.

10.2.6.2 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.

10.2.6.3 If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size.

10.2.6.4 Consult your supervisor or manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick).

10.2.6.5 Equipment blank will be generated when samples undergo particle size reduction. This blank is used to evaluate sieve cleanliness. The blank is generated after the sample goes through the sieve.

10.2.7 DETERMINATION OF APPROPRIATE EXTRACTION FLUID

10.2.7.1 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.

10.2.7.2 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.

10.2.7.3 For SPLP, refer to Section 7.10 for fluid selection. Matrix type must be specified by the client. Check special instructions or see the project manager, then record the fluid type selected on the SPLP worksheet.

10.2.7.4 The TCLP leaching fluid for all volatiles is Fluid #1.

10.2.7.5 For TCLP leach fluid determination for non-volatile analytes, continue with the following steps.

10.2.7.6 Calibrate the pH meter with fresh buffer solution in accordance with the pH SOP.

10.2.7.6.1 The following procedure is applicable for use with the Accumet AR25 pH meter.

10.2.7.6.1.1 Rinse the electrodes with reagent water and place in the pH 4.0 buffer. Enter the value (4.0) of the standard into the pH meter using the touch screen. Allow the value to stabilize.

10.2.7.6.1.2 Rinse the electrodes and place in the pH 7.0 buffer. Enter the value (7.0) of the standard into the pH meter using the touch screen. Allow the value to stabilize.

10.2.7.6.1.3 Rinse the electrodes and place in the pH 10.0 buffer. Enter the value (10.0) of the standard into the pH meter using the touch screen. Allow value to stabilize.

10.2.7.6.2 The pH meter should be calibrated daily. The calibration is recorded on the analytical log sheet. The manufacturer recommended slope is 95.0 %. If the slope is not met, pour fresh buffer solutions into the cups and try again. If this does not resolve the problem, change the KCL solution in the electrode. If still having a problem the probe may need to be changed.

10.2.7.6.3 The pH readings must be within 0.05 pH units of the buffer solution value. If not repeat adjustments on successive portions of the two buffer solutions until the readings are within 0.05 pH units (s.u.).

10.2.7.6.4 If the pH meter has been turned off, it must be calibrated prior to use.

10.2.7.7 Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a 250-mL beaker and enter the actual weight on the TCLP worksheet.

10.2.7.8 Add 96.5 ± 1.0 mL of reagent water, cover with a watchglass, and stir for 5 minutes on a stirrer and enter the actual volume on the TCLP worksheet.

10.2.7.9 Measure and record the sample pH. **Note**: To avoid damaging the pH probe when organic liquid is present, use narrow range pH indicator paper.

10.2.7.10 If the pH is less than or equal to 5.0, use Fluid #1 and proceed to Section 10.2.8 (Fluid Compatibility).

10.2.7.11 If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl, cover with a watchglass. Slurry the sample briefly then heat at 50 ^{o}C for 10 minutes. Record the temperature of the hotplate on the TCLP worksheet. **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.

10.2.7.12 Cool to room temperature.

10.2.7.13 Measure and record the pH immediately after the sample has reached room temperature.

10.2.7.13.1 If the pH is less than or equal to 5.0, use Fluid #1.

10.2.7.13.2 If the pH is greater than 5.0, use Fluid #2.

10.2.8 DETERMINATION OF FILTRATE/EXTRACTION FLUID COMPATIBILITY (skip this step for SPLP extractions)

10.2.8.1 Place 5 mL of the appropriate leaching fluid (determined in the previous step) into a 20-25 mL vial. **Note**: Use fluid type # 1 if simply testing the filtrate for a sample with less than 0.5% solids.

10.2.8.2 Add 5 mL of the initial filtrate, cap and shake.

10.2.8.3 If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation.

10.2.8.4 If the phases are NOT miscible, the initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined (see Section 11.1.4).

10.2.9 For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 10.3.

10.2.10 For samples requiring analysis for volatile organics (ZHE), proceed to Section 10.4.

10.3 BOTTLE EXTRACTION PROCEDURE: NON-VOLATILE CONSTITUENTS: SEMI-VOLATILES, PESTICIDES, HERBICIDES, METALS (Refer to Flow Chart #2, Appendix D)

10.3.1 All masses should be recorded to the nearest 0.1 g.

10.3.2 The aliquot used in the Preliminary Evaluation MAY be used for this procedure ONLY if it was not oven dried. If the sample is 100% solid or if the preliminary aliquot was not oven dried proceed directly to Section 10.3.7 (Particle Size Reduction). If the Preliminary Evaluation aliquot was oven dried then, using a fresh aliquot of sample, continue as described in Sections 10.3.3 through 10.3.6.

10.3.3 Examine the sample and determine the type of filtration to employ per Section 10.2.5.2.

10.3.4 Repeat the steps outlined in Sections 10.2.5.3 through 10.2.5.5.3.

10.3.5 Determine and record the volume (mass) of the initial filtrate. Cover with aluminum foil and retain for use as defined in Section 10.3.18.

10.3.6 Determine and record the “solid” phase mass by subtracting the mass of the liquid filtrate from the mass of the subsample.

10.3.7 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.

10.3.7.1 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 should 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.

10.3.8 Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 10.2.3.

10.3.9 Divide the total volume of solid phase leachate required by 20 to determine the mass of solid phase required for leaching. Round this mass UP to the nearest 5g.

10.3.10 Weigh the required mass of solid phase into an appropriate bottle (plastic for metals only, glass for all others) and **slowly** add 20 times its mass of appropriate leaching fluid as determined under Section 10.2.7 (e.g., 20 g of sample would require 400 g of leaching fluid). Record the weight of the sample aliquoted for the extraction on and the amount of extraction fluid added on the TCLP worksheet.

10.3.10.1 Record the vessel number used for each sample on the TCLP worksheet. Note that the same vessel should not be used more than once for the method blank, if practical.

10.3.10.2 Vessels are labeled and tracked in a logbook. The date that the vessel is first used and the date it is disposed of are included in this logbook.

10.3.11 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. The room temperature and time should be checked at both the start and end of the extraction and recorded on the TCLP worksheet. The actual tumbler speed will be monitored monthly in a Tumbler RPM 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.

10.3.12 Remove the bottle and filter the sample using vacuum or pressure filtration by filtering through a new glass fiber filter as discussed in Sections 10.2.5.5.1 - 10.2.5.5.2. 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. Record the date and time the filtration is started on the TCLP worksheet.

10.3.13 If the waste contained no initial filtrate, this solution from 10.3.12 is defined as the TCLP leachate.

10.3.14 If the waste did yield an initial filtrate, consult the worksheet for initial filtrate/leachate compatibility. If they are compatible, they are to be combined in the correct proportions (see Section 11.1.4) and mixed well. This combined solution is defined as the TCLP leachate.

10.3.15 If the individual phases are NOT compatible, they are to be prepared and analyzed separately and the results combined mathematically. See Section 11.1.5.

10.3.16 Measure and record the pH of the TCLP leachate on the TCLP worksheet. (Do not attempt to measure the pH of oily samples as the probe may be rendered inoperable.)

10.3.17 Prepare subsamples for metals for MS/MSD quality control testing using the appropriate TCLP spiking solution (do not spike for organics). Record the lot number of the spiking solution on the TCLP worksheet. Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.

10.3.18 Immediately preserve the leachate as follows:

Metals pH < 2 w/50% HNO_3 for non-oils (do not acidify oils)
All others Refrigerate to 4 ± 2 ^{o}C

Note: Refer to Section 8.6 if precipitation occurs upon preservation.

10.3.19 Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis with copies of the TCLP preparation worksheets.

10.4 ZHE EXTRACTION PROCEDURE: VOLATILE CONSTITUENTS (Refer to Flow Chart #3, Appendix D)

10.4.1 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, etc...).

10.4.2 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.

10.4.3 If the TCLP extraction is for volatile components only, refer to Section 10.2.5.1 before proceeding.

10.4.4 All masses should be recorded to the nearest 0.1 g.

10.4.5 Assemble the ZHE apparatus. Test for leakage by closing all valves except the gas inlet/outlet valve and pressurizing to 50 psi. Allow to stand for 15 minutes and check the pressure on the built-in gauge to make sure it is not leaking. If the pressure is NOT 50 psi, consult your supervisor.

10.4.6 Adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

10.4.7 Consult the worksheet and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor.

10.4.8 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. **Note:** To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 ºC) 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 10.3.11.

10.4.8.1 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 should be subcontracted out for particle size reduction (Contact PA or PM).

10.4.9 Place the ZHE apparatus on the balance and tare the balance.

10.4.10 Determine the appropriate size subsample to weigh using the percent solids information from Section 10.2.5.

10.4.10.1 For wastes that are 100% solids, a 25 g sample is used.

10.4.10.2 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.

10.4.10.3 For wastes containing ≥ 0.5% and < 5.0% solids, a 500 g subsample of waste is recommended.

10.4.10.4 If the sample has ≥ 5.0% solids, the appropriate sample size should be determined using the equation in Section 12.1.2. **Note:** For wastes containing greater than 0.5% wet or dry solids (Section 10.2.5), 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 10.2.5 may differ from the filtration solids due to sample variability or differences in the filtration apparatus.

10.4.11 Homogenize and transfer an appropriate size subsample of the waste into the ZHE and record the mass.

10.4.12 Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

10.4.13 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.

10.4.14 If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 10.4.18.

10.4.15 If the waste is < 100% solids, 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.

10.4.16 Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the pre-weighed syringe or Tedlar bag to the liquid inlet/outlet valve and open the valve. Record the tare weight of the collection device.

10.4.17 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 2 minute period. **Note**: If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe and return to Section 10.4.15.

10.4.18 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. Remove the collection device and record the total weight of the collection device with filtrate. Close the valve and discontinue gas pressure. Transfer the filtrate to VOA vials and label appropriately. Calculate the weight of filtrate collected. **Notes:** If the original waste contained less than 0.5% solids (Section 11.2.5), this filtrate is defined as the TCLP leachate and you may proceed to Section 10.4.28. Otherwise, save the vials by storing at 4 C under minimal headspace conditions, for recombination as in Section 10.4.27. The material remaining in the ZHE is defined to be the "solid" phase. Calculate the weight of the solid phase.

10.4.19 Based on the information from Sections 10.2.5 and 10.4.11 and using the formula in 11.1.3, determine the weight of fluid to load into the ZHE on the "solid" phase. The ZHE device has approximately a 500-mL capacity. Based on the need to add an amount of extraction fluid equal to 20 times the mass of the "solid" phase, the ZHE can therefore accommodate a maximum of 25 grams of "solid". **Note:** The TCLP ZHE prep uses only TCLP fluid #1; the SPLP ZHE prep uses only SPLP fluid #3

10.4.20 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.

10.4.21 Using a stainless steel syringe transfer the required volume into the ZHE and close the valve.

10.4.22 Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) 2 or 3 times. Reposition the ZHE in the vertical position.

10.4.23 Pressurize the ZHE to 5-10 PSI. Allow to stand for 10 minutes, and then recheck the pressure. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the analysis.

10.4.24 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.

10.4.25 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 and time should be checked at both the start and end of the extraction and recorded on the ZHE worksheet.

10.4.26 Confirm that the pressure of 5-10 PSI was maintained throughout the leaching. If it was NOT maintained, return to Section 10.4.1 and repeat the leachate with a new aliquot of sample.

10.4.27 Attach a syringe or Tedlar bag and open the liquid inlet/outlet valve to collect the aqueous leachate and proceed as outlined in 10.4.19 - 10.4.20. Record the volume/mass of the leachate and any oil phase. Record the date and time the filtration is completed on the ZHE worksheet. **Notes:** If the waste contained an initial liquid phase, the liquid may be filtered directly into the same collection device holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combination would create multiple phases or there is not enough volume left within the filtrate collection container.

10.4.28 If the waste contained an initial filtrate (Section 10.4.18) that is miscible with the solid phase leachate (as determined in Section 10.2.8), the solid phase leachate and the initial filtrate are directly recombined in the correct proportions (see Section 11.1.4). If the

individual phases are NOT compatible, they are to be collected, prepped and analyzed separately. **Note:** Chill the filtrate and receiving vessels before recombining.

10.4.29 Following collection, store the TCLP leachate in 2 40-mL VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic extraction procedure. Due to space limitations, the rest is disposed.

10.4.30 If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 11.1.5.

11. CALCULATIONS/DATA REDUCTION

11.1 Calculations

11.1.1 Calculation of Percent Wet Solids:

$$Percent\ \ Wet\ Solids = 100\left(\frac{Mass,"solid"\ phase}{Mass,initial\ subsample}\right)$$

11.1.2 Calculation of weight of waste to charge to ZHE:

$$Weight\ of\ \ waste\ to\ charge\ to\ ZHE = 100\left(\frac{25}{\%wet\ solids}\right)$$

11.1.3 Calculation of weight of extraction fluid to use:

$$Weight\ of\ \ Extraction\ \ fluid = \frac{20\,x\,\%\ wet\ solids\,x\ weight\ of\ \ waste\ to\ be\ extracted}{100}$$

11.1.4 Calculation of volume of initial filtrate phase to recombine with solid phase leachate:

$$\text{Volume of filtrate for recombination} = \left(\frac{Weight\ \ of\ \ solids\ \ leached}{Total\ \ weight\ \ of\ \ solids}\right)\left(\frac{Leachate\ \ recovered}{Fluid\ \ added}\right)\left(Volume\ \ of\ \ initial\ \ aqueous\ \ filtrate\right)$$

11.1.5 Mathematical recombination of analytical results:

$$Final\ Analyte\ Concentration = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

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).,

11.2 REPORTING REQUIREMENTS

11.2.1 Follow these reporting conventions for multi-phase samples:

11.2.1.1 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.

11.2.1.2 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.

11.2.1.3 If one phase is "ND" and the other phase has a positive result, use the reporting limit for the "ND" phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate that "a positive result was detected below the calculated detection limit."

11.2.2 Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

11.2.3 For limits and significant figures, consult the appropriate analytical methods.

11.2.4 Anomalies - all anomalies observed during the leach procedure must be noted on the worksheet or an anomaly form. Some examples of such anomalies are:

11.2.4.1 Sample was monolithic - subsample was obtained by crushing, cutting, grinding, sawing, etc.

11.2.4.2 Insufficient sample - less than the required 100 g minimum was available.

11.2.4.3 If less than the required minimum sample is available, the client will be contacted to determine whether or not to proceed with the extraction. If the client chooses to proceed, an explanation will be included in the Case Narrative.

11.2.4.4 Multiple phases - "X" phases were present.

11.2.4.5 Sample was oil - single phase.

11.2.4.6 Sample contained liquid that did not filter under test conditions.

11.3 REVIEW REQUIREMENTS

11.3.1 Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation form was properly documented and routed.

11.3.2 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.

11.3.3 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 for lead.

12. METHOD PERFORMANCE

12.1 Refer to individual analysis SOPs.

12.2 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.

13. POLLUTION PREVENTION

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2 This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 PT-HS-001. The following waste streams are produced when this method is carried out.

14.1.1 Remaining TCLP extracts. This waste is neutralized to a pH of 6 to 9 and discharged down a lab sink.

14.1.2 Used Solvent Waste (Methanol). This waste is collected in containers identified as "Mixed Flammable Solvent Waste", Waste #3.

14.1.3 Acid Waste. This waste is collected in containers identified as "Waste Acid", Waste #33. The waste is neutralized to a pH of 6 to 9 and discharged down a lab sink.

14.1.4 Solids including soils, filter paper, paper towels. This waste is collected in containers identified as "Lab Trash", Waste #12.

15. REFERENCES/CROSS-REFERENCES

15.1 Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update IV.

15.2 Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, September 1994, SW-846 Final Update IV.

15.3 Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.

15.4 Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.

15.5 Technical Background Document and Response To Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.

15.6 PT-IP-003: Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods.

15.7 PT-MT-001: Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, Method 6010B and Method 200.7.

15.8 PT-MT-005: Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1.

15.9 PT-MS-002: Determination of Volatile Organics by GC/MS based on Method 8260B.

15.10 PT-MS-001: GC/MS Analysis Based on Method 8270C, SW846.

15.11 PT-GC-001: Gas Chromatographic Analysis Based on Methods 8000B, SW-846 8081A, 8082, 8141A, 8151A, 610 and 8310, 8041 and 604.

15.12 PT-OP-001: Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, 8151A and 600 Series Methods.

15.13 PT-MT-002: Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS) for Methods 200.8, 6020 and ILM05.2.

15.14 PT-QA-021, TestAmerica Pittsburgh QC Program

15.15 PT-QA-024, Subsampling.

15.16 PT-QA-025, Implementation of the DoD QSM Version 3.

16. METHOD MODIFICATIONS

16.1 Modifications/Interpretations from Reference Methods

16.1.1 Section 8: Preliminary Evaluations. Section 7.1 of the source method 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 ovendrying step may not be used for TCLP extraction because solid phase degradation may result upon heating.

16.1.2 Section 11.2.5.6.3: Percent Solids Determination. Section 7.1.2 of the source method indicates that "if the percent wet solids is ≥ 0.5% and it is noticed that a small amount of the filtrate is entrained in wetting of the filter" that the filter should be oven dried to determine percent dry solids ". Drying of oil or organic matrices can both be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.

16.1.3 Section 11.2.8: Preliminary 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 and incorporated into the Preliminary Determinations section.

16.1.4 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.

16.1.5 Section 11.2.7.9: 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.

16.1.6 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 QAPP 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.

16.1.7 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.

16.2 Modifications from Previous SOP

Safety, Pollution Prevention and Waste Management Sections were updated. Worksheets were updated. pH meter calibration instructions were included.

16.3 Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must

be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17. ATTACHMENTS

17.1 Documentation and Record Management

The following documentation comprises a complete TCLP preparation raw data package:

- Completed worksheets (Appendix C).
- Non-conformance summary (if applicable).
- Anomaly documentation (if applicable).

18. REVISION HISTORY

18.1 Revision 4:

18.1.1 Updated the SOP to the new corporate format; updated section and SOP reference numbers; changed STL to TestAmerica throughout the SOP; added corporate text to the Scope, Safety, Pollution Control and Waste Management sections.

18.1.2 Added text to section 10.13.11 concerning the Tumbler/ZHE RPM logbook; added or updated the worksheets to reflect the TestAmerica logo.

18.1.3 Added to section 10.2.6.5: Equipment blank will be generated when samples undergo particle size reduction. This blank is used to evaluate sieve cleanliness. The blank is generated after the sample goes through the sieve.

18.1.4 Section 10.4.21, added use of stainless steel syringe to transfer samples.

APPENDIX A - TABLES

APPENDIX A

TABLES

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

APPENDIX A - TABLES

Contaminant	mg/L
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

APPENDIX B

FIGURES

APPENDIX B - FIGURES

Figures 1 & 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

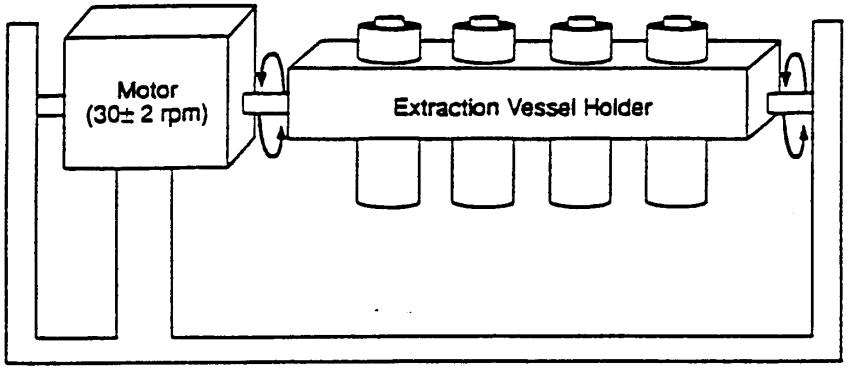
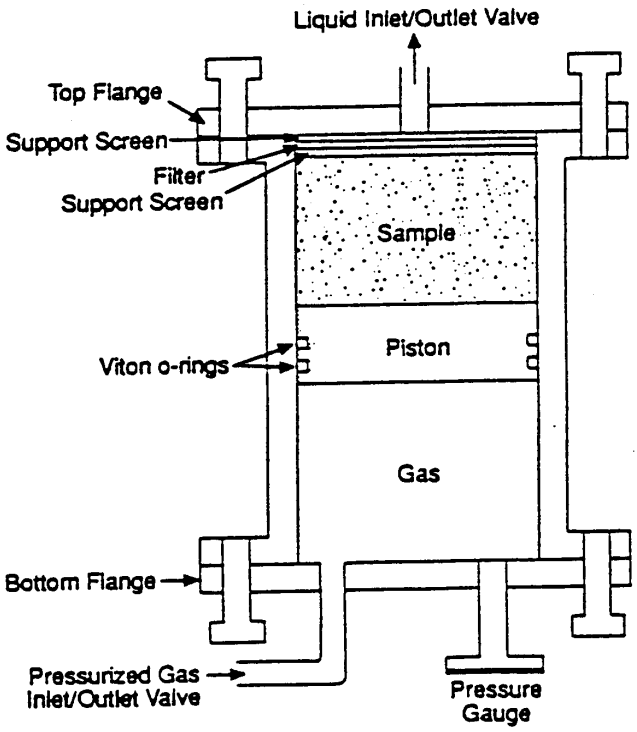


Figure 1. Rotary Agitation Apparatus



APPENDIX B - FIGURES

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.

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APPENDIX B - FIGURES

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10

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

EXAMPLE WORKSHEETS/LOGBOOKS

APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS

TestAmerica Pittsburgh BOOK ID: WC2484

TCLP Multiphasing Worksheet

"Sample volumes used for TCLP Recombination of Multiphase samples"

Analyst:_____

Date of Prep:_____

Reviewed By Analyst:_____

LOT#:_____

SAMPLE ID#_____Volume_____(ml)(Top Layer)

SAMPLE ID#_____Volume_____(ml)(Bottom Layer)

Analyst:_____

Date of Prep:_____

Reviewed By Analyst:_____

LOT#:_____

SAMPLE ID#_____Volume_____(ml)(Top Layer)

SAMPLE ID#_____Volume_____(ml)(Bottom Layer)

printed on: Monday, November 19, 2007 5:10:15 PM Page 1 of 1

APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS

(TestAmerica PITTSBURGH) TCLP Preperation Worksheet -- Nonvolatile Extraction

Workorder: _____ Date of Prep: _____

Client: _____ Prepped by: _____

Reviewed by: _____

Sample Lot ID: _____

I. Preliminary Investigation:

Sample Description:

_____

_____

_____

_____

_____

II. Determination of Percent Solids: (Not required if sample obviously will not Yield a liquid under pressure; assume 100% solids)

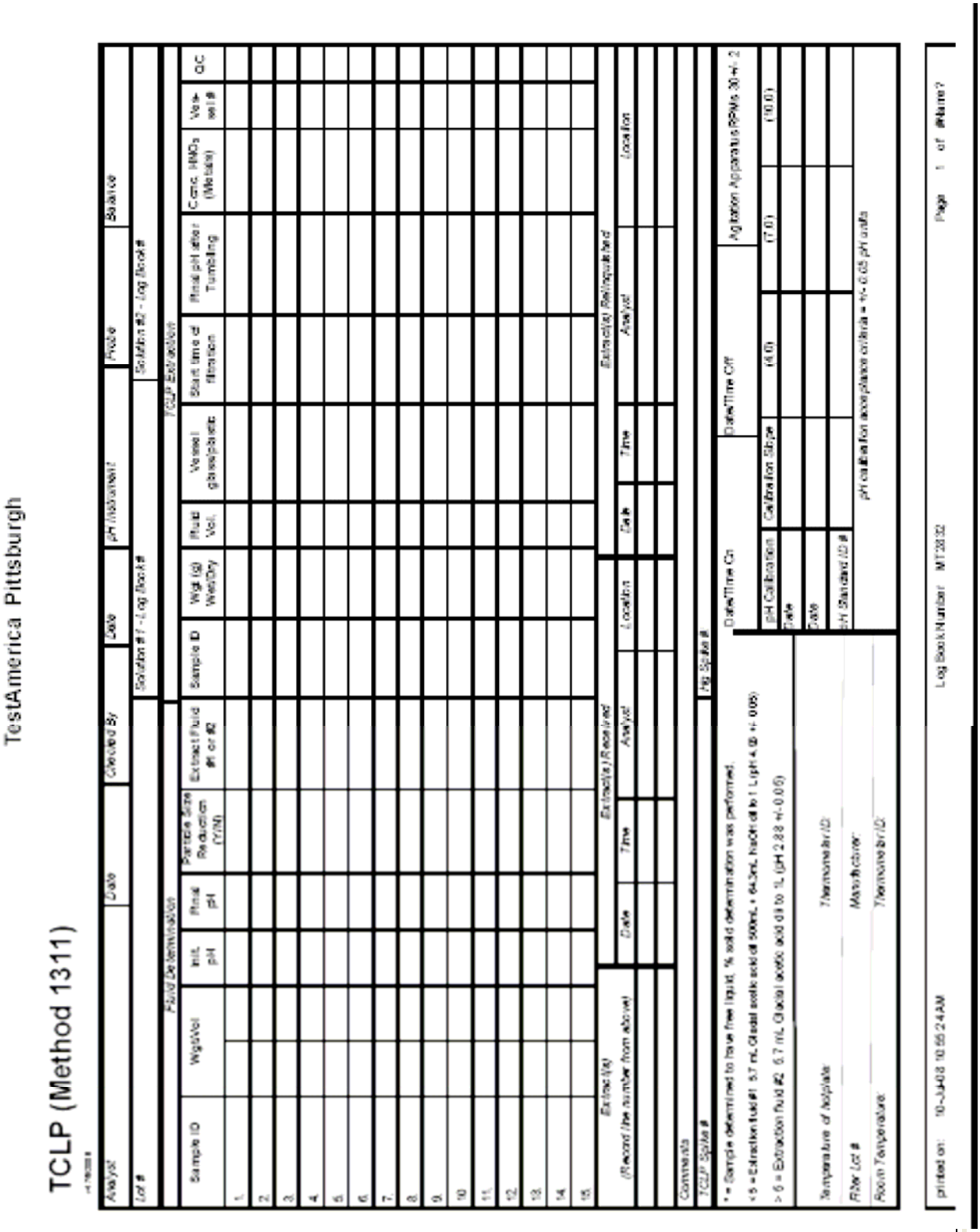
Weight of Sample container before pouring into filter:		g.
- Weight of Sample container after pouring into filter:		g.
Weight of sample used (under ideal conditions):	0.0	g.
Weight of Beaker and Filtrate after filtering:		g.
- Weight of Beaker (empty) before filtering:		g.
Weight of FILTRATE Collected:	0.0	g.
Weight of Filter paper, screen & Solids after filtering:		g.
- Weight of Filter paper & screen before filtering:		g.
Weight of SOLIDS Collected:	0.0	g.

Percent Solids (actual): #DIV/0!

Percent Solids (ideal): #DIV/0!

Percent loss of material (ideal vs. actual): #DIV/0!

APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS



TestAmerica Pittsburgh

TCLP (Method 1311)

Analyst | Date | Checked By | Date | pH Instrument | Probe | Balance

Lot # | Solution #1 - Log Book # | Solution #2 - Log Book #

Fluid Determination | TCLP Extraction

Sample ID	Wgt/Vol	Init. pH	Final pH	Particle Size Reduction (Y/N)	Extract Fluid #1 or #2	Sample ID	Wgt (g) Wet/Dry	Fluid Vol.	Vessel glass/plastic	Start time of filtration	Final pH after Tumbling	Conc. HNO3 (Metals)	Ver-sel #	QC
1.														
2.														
3.														
4.														
5.														
6.														
7.														
8.														
9.														
10.														
11.														
12.														
13.														
14.														
15.														

Extract(s) Received | Extract(s) Relinquished

Extract(s) (Record the number from above) | Date | Time | Analyst | Location | Date | Time | Analyst | Location

Comments

TCLP Spike # | Lig Spike #

* = Sample determined to have free liquid, % solid determination was performed

< 5 = Extraction fluid #1 5.7 mL Glacial acetic acid at 500mL + 64.3mL NaOH dil to 1 L (pH 4.93 +/- 0.05)

> 5 = Extraction fluid #2 5.7 mL Glacial acetic acid dil to 1L (pH 2.88 +/- 0.05)

Date/Time On | Date/Time Off | Agitation Apparatus RPMs 30 +/- 2

pH Calibration	Calibration Slope	(4.0)	(7.0)	(10.0)
Date				
Date				
pH Standard ID #	pH calibration acceptance criteria = +/- 0.05 pH units			

Temperature of hotplate: Thermometer ID:

Rinse Lot # Manufacturer:

Room Temperature: Thermometer ID:

printed on: 10-Jul-08 10:55:24 AM | Log Book Number MT2832 | Page 1 of #Name?

APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS

TestAmerica Pittsburgh (SPLP-Method 1312) LEACHATE LOGBOOK Logbook ID: OP2472

Analyst	Date	Reviewed by	Date	pH Instrument	Probe	Balance

Lot Number(s)	Solution #1 - Logbook Number	Solution #2 - Logbook Number

Work Order #	Comments	Init pH	Extract Fluid #1, #2 or #3	Weight (gm) Wet / Dry	Fluid Volume	Vessel Glass / Plastic	Final ph after tumbling	Conc HNO3 to Acidify (for Metals)
1.								
2.								
3.								
4.								
5.								
6.								
7.								
8.								
9.								
10.								
11.								
12.								
13.								
14.								
15.								
16.								
17.								
18.								
19.								
20.								
21.								
22.								

Extract(s)	Extract(s) Received				Extract(s) Relinquished			
(record line # from above)	Date	Time	Analyst	Location	Date	Time	Analyst	Location

* = Sample determined to have free liquid, % solid determination was performed

Extraction Fluid 1 = Samples that are EAST of the Mississippi River. Prepared by adding an aliquot of a 60/40 % by weight mixture of H_2SO_4 and HNO_3 to reagent water (pH 4.20 +-0.05)

Extraction Fluid 2 = Samples that are WEST of the Mississippi River. Prepared by adding an aliquot of a 60/40 % by weight mixture of H_2SO_4 and HNO_3 to reagent water (pH 5.00 +-0.05)

Extraction Fluid 3 = This is a reagent water and is used to determine cyanide leachability

Date/Time on		Date/Time off		Agitation Apparatus RPM: 30 +- 2	
	(4.0)		(7.0)		(10.0)
Date					
Date					

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APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS

TestAmerica Pittsburgh

ZHE (Method 1311) LEACHATE LOGBOOK

Logbook ID: MV2506

Analyst	Date	Lot #	Probe
Checked By	Date	Matrix:	pH Instrument
Solution #1 - Log Book #	Room Temp.		Batch #

Lab Smp ID #	Client ID	Vessel	Weight (gm)	Extraction Fluid No. / Volume	ZHE Pressure Initial	ZHE Pressure Final	pH after tumbling
1.							
2.							
3.							
4.							
5.							
6.							
7.							
8.							
9.							
10.							
11.							
12.							
13.							
14.							
15.							
16.							
17.							
18.							
19.							
20.							
21.							
22.							
23.							

Extract(s) (Record the number from above)	Extract(s) Relinquished Date	Time	Analyst	Location	Extract(s) Received Date	Time	Analyst	Location

* = Sample determined to have free liquid, % solid determination was performed
<5 = Extraction fluid No. 1 5.7ml Glacial Acetic Acid dil 500mL + 64.3mL of 1 N NaOH dil to 1 L pH 4.93 + 0.05)

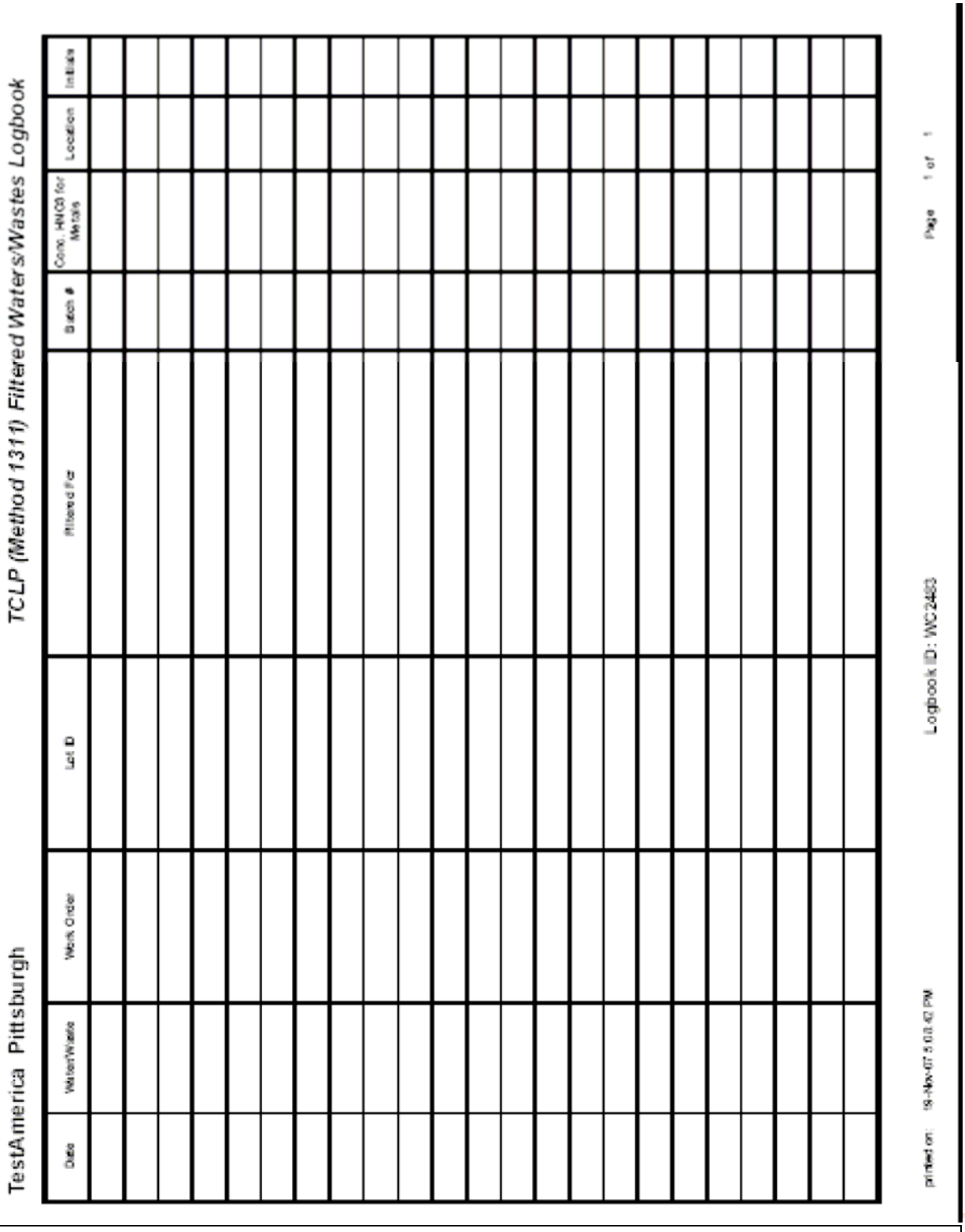
pH Calibration Date	Calibration Slope	(4.0)	(7.0)	(10.0)	
pH Standard ID					
Filter Lot ID #			Balance ID #		

Date/Time On	Agitation Apparatus RPM's 30±2 checked by
Date/Time Off	

printed on: 20-Nov-07 10:19:25 AM

Page 1 of 1

APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS



TestAmerica Pittsburgh

TCLP (Method 1311) Filtered Waters/Wastes Logbook

Date	Water/Waste	Work Order	Lot ID	Filtered For	Batch #	Conc. HNO3 for Metals	Location	Initials

printed on: 19-Mar-07 5:03:42 PM

Logbook ID: WC2483

Page 1 of 1

APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS

TestAmerica Pittsburgh ***EXTRACTIONS*** LOGBOOK ID: OP2914

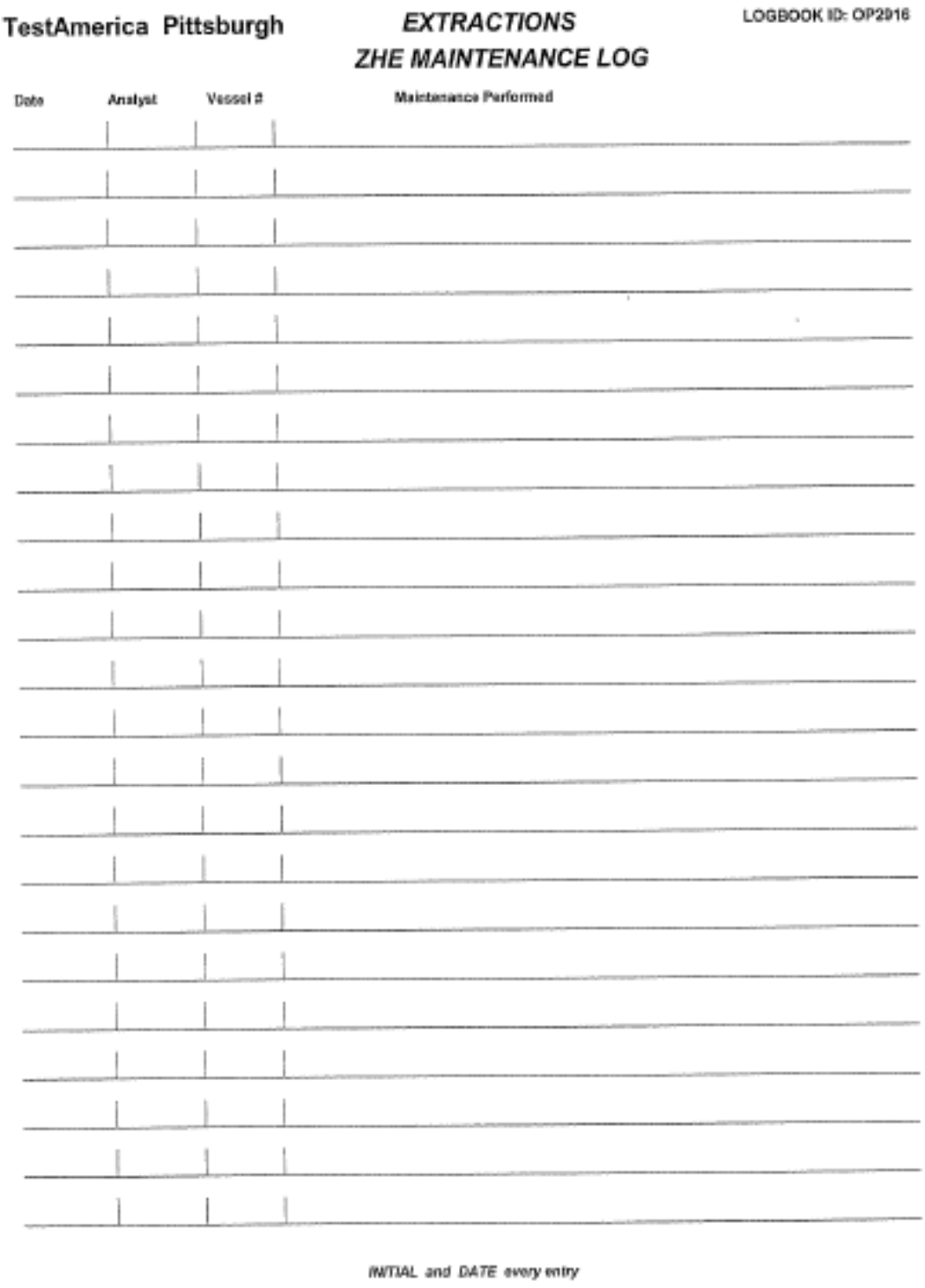
TCLP VESSEL MAINTENANCE LOG

Date	Analyst	Vessel #	Maintenance Performed

INITIAL and DATE every entry

printed on: 23-Sep-08 9:09:26 AM Page 1 of 1

APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS



TestAmerica Pittsburgh ***EXTRACTIONS*** LOGBOOK ID: OP2916

ZHE MAINTENANCE LOG

Date	Analyst	Vessel #	Maintenance Performed

INITIAL and DATE every entry

printed on: 23-Sep-08 9:11:00 AM Page 1 of 1

APPENDIX C – EXAMPLE WORKSHEETS/LOGBOOKS

TestAmerica Pittsburgh *TCLP/ZHE Tumbler RPM Log* Logbook ID: OP2892

MONTH: YEAR:

Location: Pittsburgh

Tumbler ID/ZHE ID: Tumbler 1

Tolerance Limit: +- 2 RPMs

Analyst Initials	Date	Time	RPM

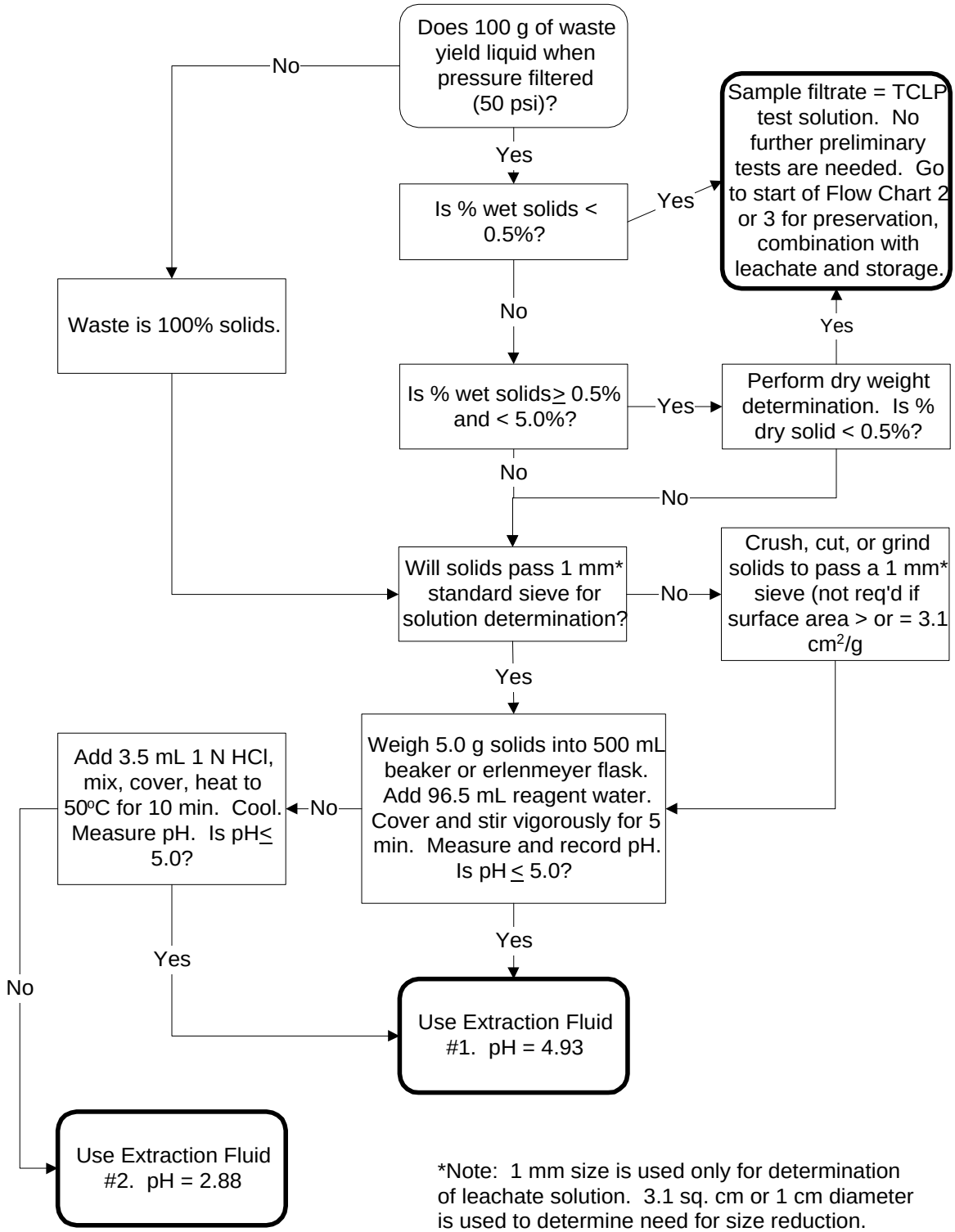
printed on: 22-Sep-08 10:58:48 AM Page 1 of 1

APPENDIX D

FLOW CHARTS

APPENDIX D – FLOW CHARTS

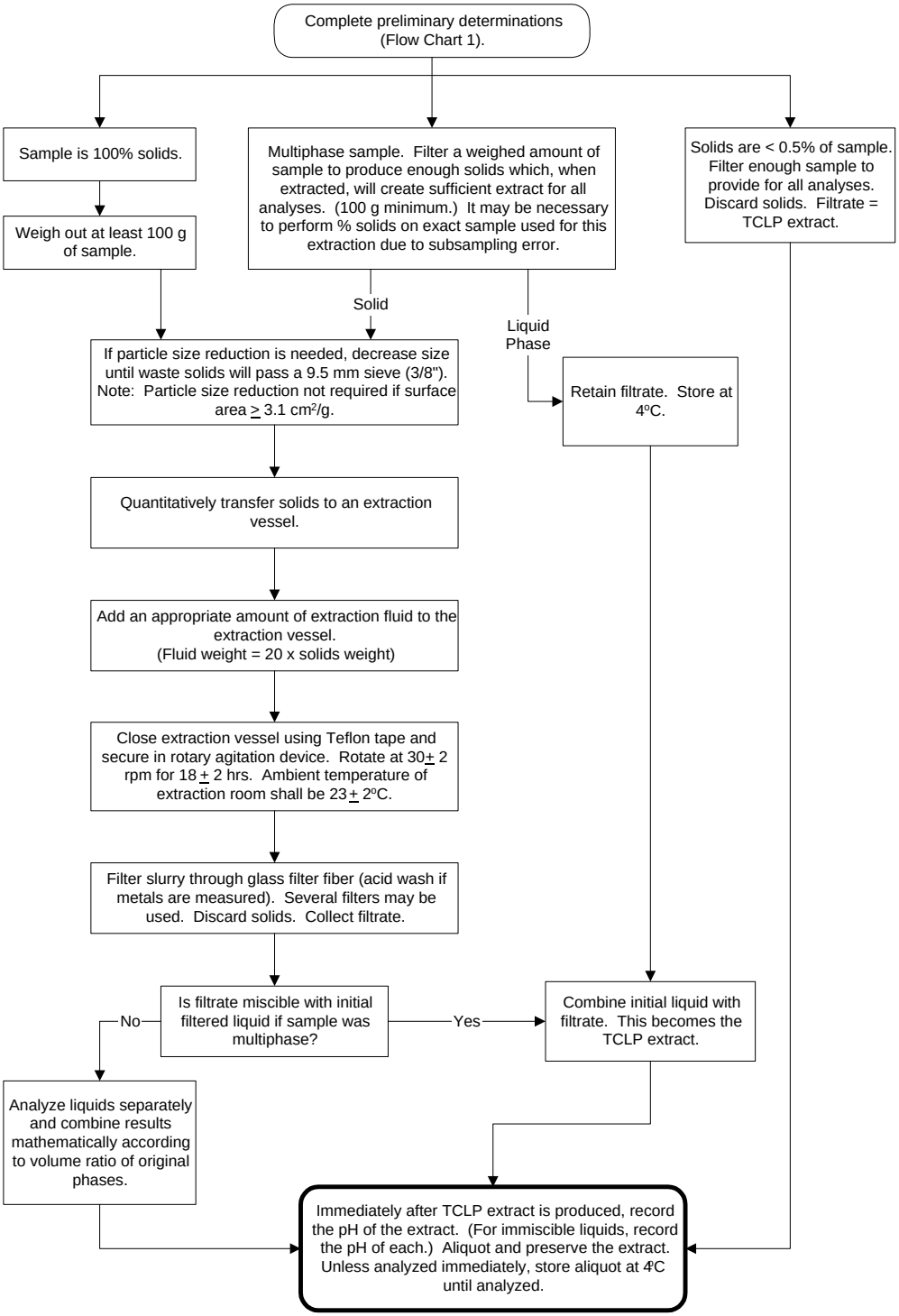
Flow Chart 1. Preliminary Sample Evaluation (Section 10.2)



*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.

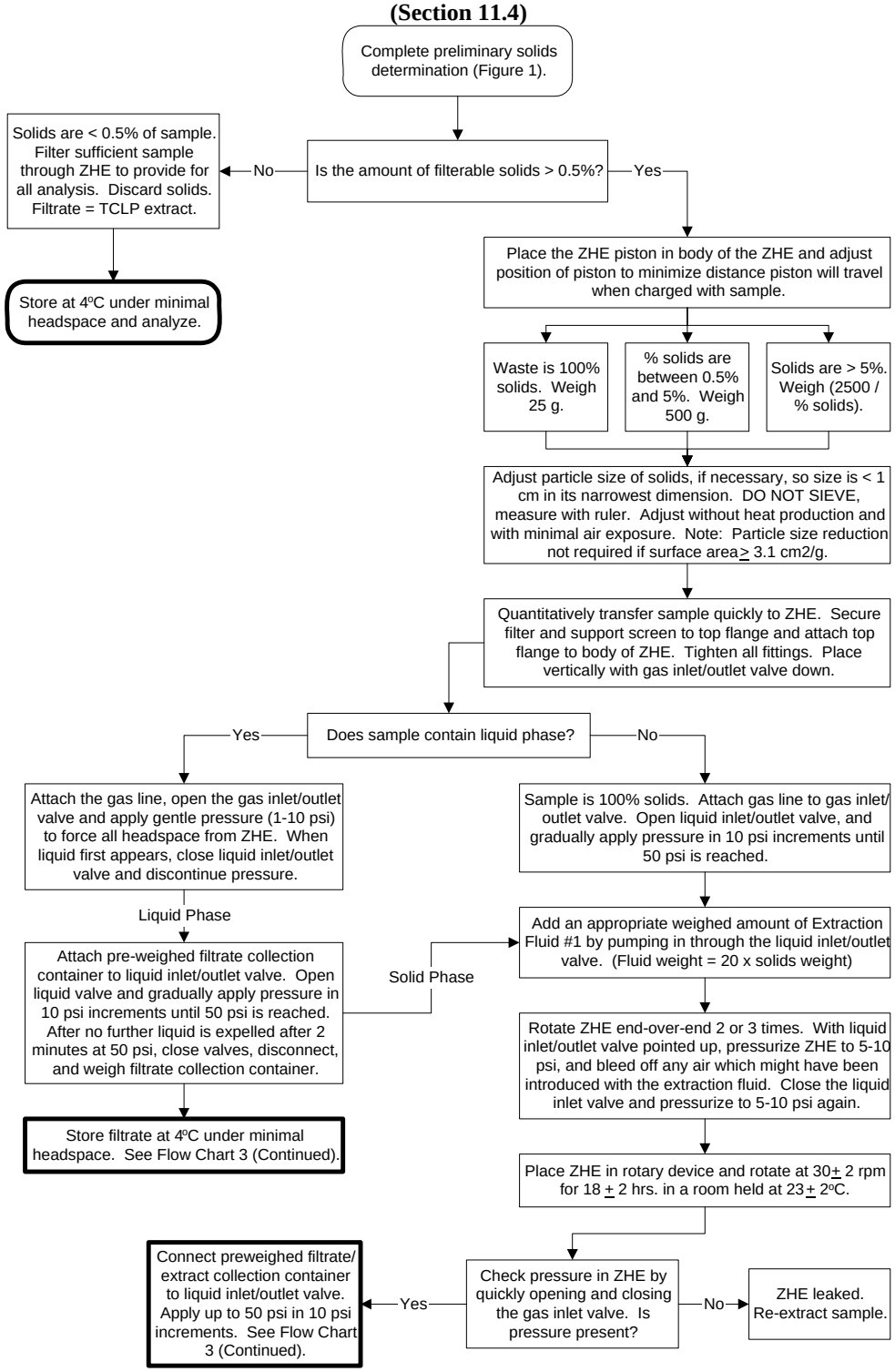
APPENDIX D – FLOW CHARTS

Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



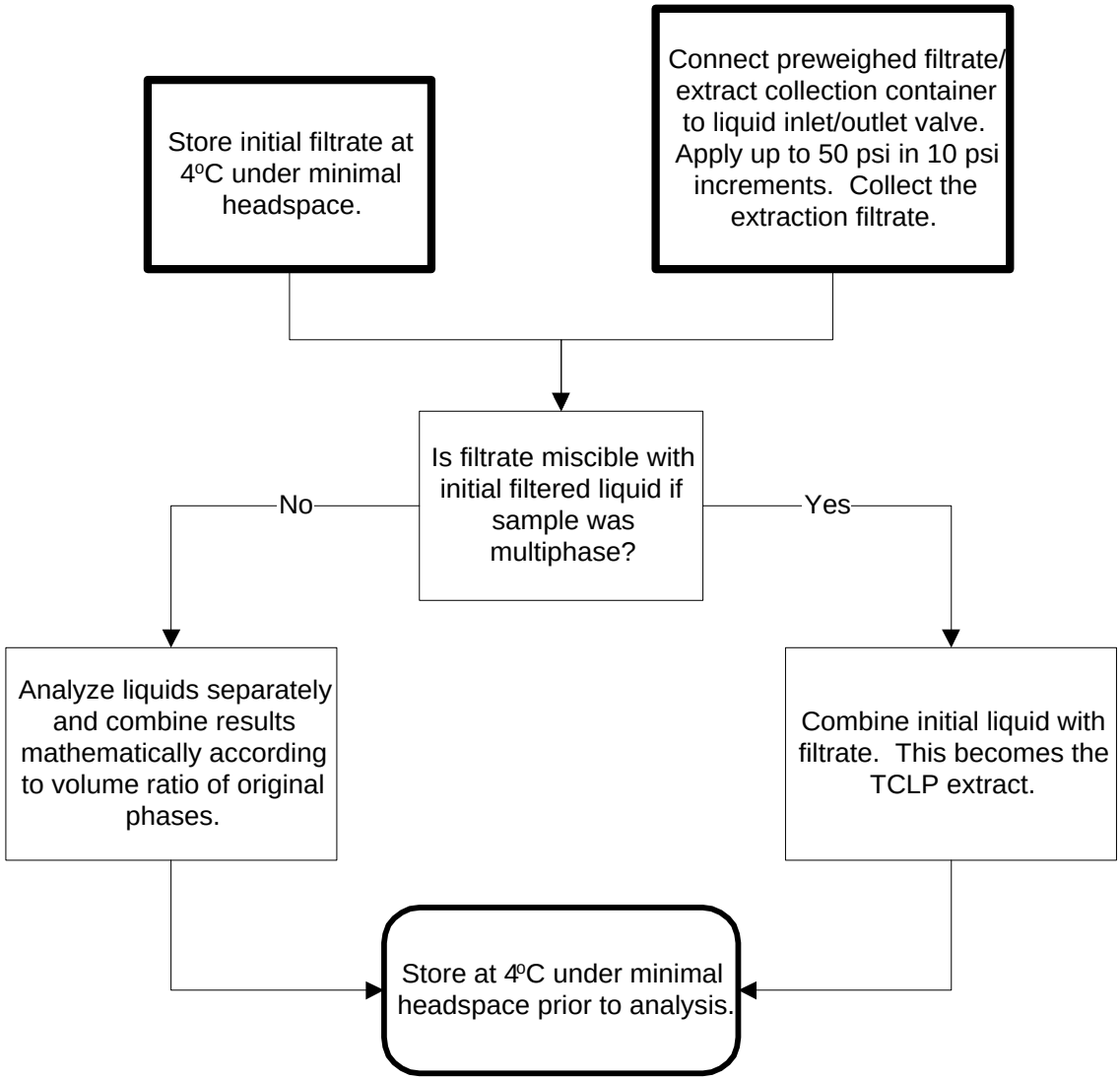
APPENDIX D – FLOW CHARTS

Flow Chart 3. ZHE Extraction, Volatile Constituents (Section 11.4)



APPENDIX D – FLOW CHARTS

Flow Chart 3. ZHE Extraction
(Continued)



TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TITLE: Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP)

METHODS: SW-846 6010B, 6010C AND EPA 200.7

Approvals (Signature/Date):

	07/17/09		07/20/09
William Reinheimer Technical Manager	Date	Steve Jackson Health & Safety Manager / Coordinator	Date
	07/07/09		07/17/09
Nasreen K. DeRubeis Quality Assurance Manager	Date	Larry Matko Laboratory Director	Date

Controlled Source: Intranet

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, 6010C and EPA Method 200.7. Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B/6010C and 200.7. Additional elements may be analyzed under Methods 6010B, 6010C and 200.7 provided that the method performance criteria presented in Section 13.0 are met. In addition to SOW ILMO4.0, this SOP is also compliant with the requirements of CLP SOWs 7/88, 3/90, ILMO1.0 and ILMO2.1.

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. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.3. Method 6010B and 6010C are applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, tissues, wipes 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 and 50 mg/kg in solid matrix samples.

1.5. For DoD QSM Version 3 requirements, refer to SOP PT-QA-025. For DoD V4.1 refer to SOP PT-QA-029.

1.6. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 12.1 in the Quality Assurance Manual.

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 a 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. Consult the appropriate SOP's for details on sample preparation methods.

2.2. Refer to the appropriate SOPs 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. 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.

4.1.2. 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.

4.1.3. 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.

4.1.4. 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.

5. SAFETY

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

5.2 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

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.			

5.4 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 RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.6 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. Metals digestates can be processed outside of a fume hood. 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 or well-ventilated area.

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 a laboratory supervisor or EH&S coordinator.

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. Nitrogen or argon gas supply, welding grade or equivalent.

6.4. Cool flow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom TestAmerica 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. 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. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix.

7.3. 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.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. 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, SHIPMENT 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 and wipe samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation. Tissue samples are stored frozen until preparation.

8.4. Dissolved metals samples that are filtered and preserved at the laboratory with concentrated Nitric acid will be held for 24 hours before digestion.

9. QUALITY CONTROL

9.1. Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action. See SOP PT-QA-021"TestAmerica Quality Control Program" for additional detail on criteria and corrective actions.

9.2. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 12.0.

9.3. Method Blank (MB) - One method blank must be processed with each preparation batch of up to 20 samples. 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 10X higher than the blank

contamination level). **Refer to PT-QA-025 for specific DoD requirements for the method blank. For DoD V4.1 refer to SOP PT-QA-029.**

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) 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 taken in consultation with the client and 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 taken in consultation with the client and 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 and the client must be notified.**
- 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) - One aqueous LCS (referred to as a Laboratory Fortified Blank in 200.7) must be processed with each preparation batch of up to 20 samples. 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. Control limits are established, for method 6010B/6010C, a control limit of 80 - 120% (85-115% for 200.7) 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 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 120% (115% for 200.7) and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and 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.

9.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch of up to 20 samples (6010B and 6010C) or one MS for every 10 or fewer samples (200.7). A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added (referred to as a Laboratory Fortified Matrix in 200.7). 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/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 method 6010B and 6010C, control limits of 75 - 125% (70 – 130% for 200.7) recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. **Refer to PT-QA-025 for specific DoD requirements for the MS.** For DoD V4.1 refer to SOP PT-QA-029. 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 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.
- For dissolved metals samples by 200.7, which have not been digested, a MS must be performed per every 10 or fewer samples by spiking an aliquot of the sample at the levels specified in Table III (Appendix A).
- For Methods **6010B/ 6010C/DoD** samples- If the MS/MSD recoveries are unacceptable, the same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. Otherwise, another sample from the same preparation batch should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% (for Method 6010C) of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. The spike recovery from the post digestion spiked sample **for method 6010B/DoD** the spike recovery should be within the range 75-125%. If this spike fails, then the dilution test (Sec. 9.6 should be run on this sample. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.

9.6 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 (1:5) 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 MDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.7 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 readings of four exposures of the ICV standard. For Method 6010B and 6010C, the ICV must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of three) 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. **Refer to PT-QA-025 for specific DoD requirements for the ICB. For DoD V4.1 refer to SOP PT-QA-029.** 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. This standard is equivalent to the Quality Control Standard (QCS) and the first Instrument Performance Check (IPC) specified in 200.7.

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 is 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 three) exposures. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. **Refer to PT-QA-025 for specific DoD requirements for the CCB.** For DoD V4.1 refer to SOP PT-QA-029. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration are not required before continuation of the run. If a mid-run CCV or CCB fails, the analytical run may be continued; however, the result(s) for the affected element(s) may only be reported when bracketed by valid CCV/CCB pairs. If analytical results for one or more elements are not bracketed by valid CCV/CCB pairs, the problem must be corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed for those elements only.

9.9 Reporting Limit Verification Standard (RLV)/CRA or LLICV/LLCCV (6010C) – Calibration accuracy at the laboratory reporting limit is verified after the analysis of the ICB by running the RLV, CRA or LLICV/LLCCV. For method 6010C it must be analyzed at beginning and end of the analytical run. This standard is at the reporting limit. For Methods 200.7 and 6010B the control limit is 50 – 150%. For Method 6010C LLICV/LLCCV the control limit is 70-130%. For method 6010C the RLV/CRA which is the low level quantitation check sample is prepared and analyzed quarterly or as needed. The control limit is 70-130%. Please note the RLV/CRA (undigested) is still analyzed at the beginning and end of the analytical run. **Refer to PT-QA-025 for specific DoD requirements for the RLV standard.** For DoD V4.1 refer to SOP PT-QA-029.

9.10. 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 TestAmerica 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 over correction as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.10.3. 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.

9.10.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 10.11 for required run sequence).

9.10.2. 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.

9.10.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the TestAmerica guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs > 10 µg/L must fall within the TestAmerica guidelines of ± 1x RL from zero. **Refer to PT-QA-025 for specific DoD requirements for the ICSA.** For DoD V4.1 refer to SOP PT-QA-029. 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.

9.12 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 10.15 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.13 Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC, which may supersede this SOP's requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understands the special project requirements.

10. PROCEDURE

10.1. Calibration and Standardization

10.1.1. 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).

10.1.2. The instruments are profiled and calibrated according to the manufacturer's recommended procedures. Thermo has set up the ICP 61E to be profiled on Cu and the Trace ICPs are to be profiled on As. All other lines are preset by Thermo and should not be adjusted by the user. Flush the system with the calibration blank. The calibration curve must consist of a minimum of a blank and a standard. Refer to the facility-specific instrument SOP or ICP instrument manual for a detailed set up and operation protocols.

10.1.3. 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.

10.1.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions. The NELAC requirement for verification of the initial calibration at varied concentrations is met daily since the ICVs, CCVs, and RLVs/CRA are all at different concentrations.

10.2. 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.

If the above criteria are met, the dissolved samples can be analyzed directly after an appropriate amount of 1:1 nitric acid is added to an aliquot of sample to adjust the acid concentration to approximately a 1% (v/v) nitric acid solution. Allowance for sample dilution should be made in the calculation.

10.3. A minimum of three exposures 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.

10.4. 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 10.1.2 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

10.5. The use of an autosampler for all runs is strongly recommended.

10.6. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV, CCV, RLV/CRA/LLICV/LLCCV), blanks (ICB, CCB, PB), interference checks (ICSA, ICSAB) and field samples (linear range) to improve the data review process.

10.7. 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.

10.8. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run. If any digestate for Method 200.7 has silver detected above 100 mg/L, add 1.0 ml of concentrated HCl to the digestate, mix and reanalyze. If the second analysis yields a higher value for silver, the second analysis is reported and discussed in the report narrative.

10.9. The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful in overcoming matrix interferences especially in high solids matrices.

However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1) peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

10.10. The use of an internal standard is **required** on any axial ICP unless the calibration and QC standards are matrix matched to each digestion procedure. The following procedural guidelines must be followed when using an internal standard:

10.10.1. Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

10.10.2. 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 should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

10.10.3. The concentration of the internal standard should 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.

10.10.4. The internal standard raw intensity counts must be printed on the raw data.

10.10.5. 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).

10.10.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

10.10.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

(1) Diluted and reanalyzed;

(2) The IS concentrations must be raised; or

(3) A different internal standard must be used.

10.11. The following analytical sequence must be used for Methods 6010B/6010C and 200.7:

Instrument Calibration

ICV

ICB

RLV/CRA/LLICV

ICSA

ICSAB

7 Samples

CCV

CCB

10 samples

CCV

CCB

Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run

CCV

CCB

RLV/CRA/LLCCV

CCV

CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B/6010C and 200.7 quality control criteria.

10.12. Full method required QC must be available for each wavelength used in determining reported analyte results.

10.13. Guidelines are provided in the appendices on procedures to minimize contamination of samples and **standards, preventive maintenance and troubleshooting.**

10.14. 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 interelement 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.

10.15. For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

- recovery of the analyte in the matrix spike is not at least 50%,
- the concentration of the analyte does not exceed the regulatory level, and,
- 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 D provides guidance on performing MSA analyses.

10.16. 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. If contractually required, the client shall be notified by the Project Manager.

10.17. Nonconformance documentation shall be filed in the project file.

10.18. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11. DATA ANALYSIS AND CALCULATIONS

11.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

11.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

11.3. RLV/CRA percent recoveries are calculated using the same equation as the ICV or CCV (replace ICV or CCV with RLV/CRA in the above equations).

11.4. 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

11.5. 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

11.6. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{CxV1xD}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout

D = Instrument dilution factor

V1 = Final volume in liters after sample preparation

V2 = Initial volume of sample digested in liters

11.7. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg/Kg, dryweight = \frac{CxVxD}{WxS}$$

Where:

C = Concentration (mg/L) from instrument readout

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.

11.8. The final concentration determined in digested wipe samples is calculated as follows:

ug/wipe = (C x V x D x 1000)

Where:

C = Concentration (mg/L) from instrument readout

V = Volume of digestate (L)

D = Instrument dilution factor

11.9. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

11.10. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} x100$$

Where:

I = Sample result (Instrument reading)

S = Dilution test result (Instrument reading × 5)

11.11. Appropriate factors must be applied to sample values if dilutions are performed.

11.12. Sample results should be reported with up to three significant figures in accordance with the TestAmerica significant figure policy.

12. METHOD PERFORMANCE

12.1. Initial Demonstration of Capability

12.1.1. An initial demonstration of capability for each method must be performed prior to analyzing samples. For the standard analyte list, the initial demonstration consists of the preparation and analysis of an LCS sample containing all of the standard analytes for the method, as well as a method detection limit (MDL) study (described below).

12.1.2. Four LCS samples are analyzed with the same procedures used to analyze samples, including sample preparation.

12.1.3. The mean recovery and standard deviation are calculated for each analyte of interest. These results are compared with the established or project-specific acceptance criteria. All four results must meet acceptance criteria before the method can be used to analyze samples. For further detail refer to SOP PT-QA-001.

12.2. Prior to analysis of any analyte using either Method 200.7, Method 6010B or 6010C, the following requirements must be met.

12.2.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client 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 determined in accordance with 40 CFR Part 136 Appendix B requirements as

detailed in TestAmerica QA SOP PT-QA-007. The result of the MDL determination must be below the TestAmerica reporting limit (RL).

12.2.2. Instrument Detection Limit (IDL) 200.7/6010B - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL will be determined quarterly (every 3 months). If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements per day. 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 IDL must be < MDL.

12.2.3. Instrument detection limits (IDLs) – Method 6010C – IDLs are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified according to the guidance in Sec. 9.9.

12.2.4. IDLs in µg/L can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement should 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 normally performed between the analysis of separate samples). IDLs should be determined at least every three months.

12.2.4.1. **DoD samples cannot be analyzed without a valid IDL**.

12.2.4.2. **For DoD, the established IDL must be less than the MDL for each analyte**.

12.2.5. Linear Range Verification (LR) - The linear range will be determined on a quarterly basis for each analyte wavelength used on each instrument as per CLP requirements. Method 6010B/6010C require that the linear range only be determined semiannually. Method 200.7 requires linear ranges to be determined annually. The standards used to define the linear range limit

must be analyzed during a routine analytical run. 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 determined concentration of the linear range standards must be within 5% of the true value. The linear range is the concentration above which results cannot be reported without dilution of the sample. If the instrument is adjusted in any way that may affect the LR's, the LR's must be redetermined. The LR data must be documented and kept on file.

12.2.6. 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 facility-specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

12.2.7. 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., GFAA or 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 facility specific instrument operation SOP and 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. 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 observed 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.

12.2.8. Rinse Time Determination - Rinse times must be determined whenever a new instrument is set up. . To determine the appropriate rinse time for a particular ICP system, the linear range verification standard 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. Linear Range Verifications are performed at a minimum of every six months. Whenever Linear Range Verifications are performed the suitability of the rinse time settings will be evaluated and the rinse time determination will be repeated when necessary.

12.3. Method performance is determined by the analysis of matrix spike and matrix duplicate samples as well as preparation blanks and laboratory control samples. The matrix spike recovery should fall within +/- 25 % and the matrix duplicates should compare within 20% RPD. Preparation blanks must meet the criteria specified in Section 9.3. The LCS should recover within 20% of the true value for aqueous LCS and within the control limits supplied by the manufacturer of the soil LCS.

12.4. Training Qualification:

12.4.1. 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.

13. POLLUTION PREVENTION

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution.

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 CW-E-M-001. The following waste streams are produced when this method is carried out.

14.1.1. Acid waste consisting of sample and rinse solution, this waste is collected in waste containers identified as "Acid Waste", Waste #33.

14.1.2. Expired Metals Standards – This waste is collected in containers identified as "Acid Waste with Metals", Waste #6.

15. REFERENCES/CROSS REFERENCES

15.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

15.2. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Method 6010B,Revision 2, December 1996.

15.3. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update IV, Method 6010C, Revision 3, February, 2007.

15.4. 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.

15.5. Standard Methods 20th Edition 2340B; Hardness by Calculation.

15.6. SOP PT-QA-001, Employee Orientation and Training, current version.

15.7. SOP PT-HS-001, Waste Collection, Accumulation and Storage, current version.

15.8. SOP PT-QA-007, Determination of Method Detection Limits (MDLs), current version.

15.9. SOP PT-QA-009, Rounding and Significant Figures.

15.10. SOP PT-QA-016, Nonconformance & Corrective Action System, current version.

15.11. SOP PT-QA-018, Technical Data Review Requirements, current version.

15.12. SOP PT-QA-021, Quality Control Program, current version.

15.13. SOP PT-QA-025, Implementation of the DoD QSM Version 3, January 2006, current version.

15.14. SOP PT-QA-029, DoD QSM Version 4.1 Requirements, current version.

16. METHOD MODICATIONS

16.1. Modifications/Interpretations from reference method

16.1.1. Modifications/interpretations from both Methods 6010B and 200.7.

16.1.1.1. TestAmerica laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

16.1.1.2. 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 has adopted the procedure in EPA CLP ILM04.1.

16.1.1.3. 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 laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, TestAmerica 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 labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. TestAmerica 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.

16.1.2. Modifications from Method 200.7.

16.1.2.1. 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.

16.1.2.2. 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.

16.1.2.3. 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. TestAmerica Pittsburgh follows the CLP ICS procedures because it is applicable to a wider number of programs. Therefore, we feel it is a more conservative approach.

16.1.2.4. 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 has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.8 provides the detailed corrective action criteria that must be followed. **Refer to PT-QA-025 for specific DoD requirements for the CCB.** For DoD V4.1 refer to SOP PT-QA-029.

16.1.3. Modifications from Method 6010B.

16.1.3.1. 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. **Refer to PT-QA-025 for specific DoD**

requirements for the method blank. For DoD V4.1 refer to SOP PT-QA-029.

16.1.3.2. 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 has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.8 for a detailed description of the required corrective action procedures. **Refer to PT-QA-025 for specific DoD requirements for the calibration blanks.** For DoD V4.1 refer to SOP PT-QA-029.

17. ATTACHMENTS

17.1. Documentation and Record Management

- The following documentation comprises a complete ICP raw data package:
- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

17.2. FIGURE 1 – FLOW DIAGRAM

17.3. APPENDIX A - TABLES

17.4. APPENDIX B - TESTAMERICA ICP DATA REVIEW CHECKLIST

17.5. APPENDIX C - TROUBLESHOOTING GUIDE

17.6. APPENDIX D - CONTAMINATION CONTROL GUIDELINES

17.7. APPENDIX E - PREVENTIVE MAINTENANCE

18. REVISION HISTORY

18.1. Revision 10, 03/31/09

18.1.1. Updated into the new Corporate format; added the appropriate Corporate text to the Scope, Safety, Waste Management and Pollution Control sections; added text to section 9 concerning the Initial Demonstration of Capability being moved to section 12; changed STL to TestAmerica throughout the SOP; updated reference numbers and SOP references throughout the document; updated the Reference section SOP numbers; in section 9.3 changed 20X to 10X; in section 10.10 changed the Trace to any axial ICP.

18.1.2. 6010C method reference added. SOP references updated.

18.1.3. Updated SOP for requirement of Method 6010C, see SOP areas highlighted referring to 6010C.

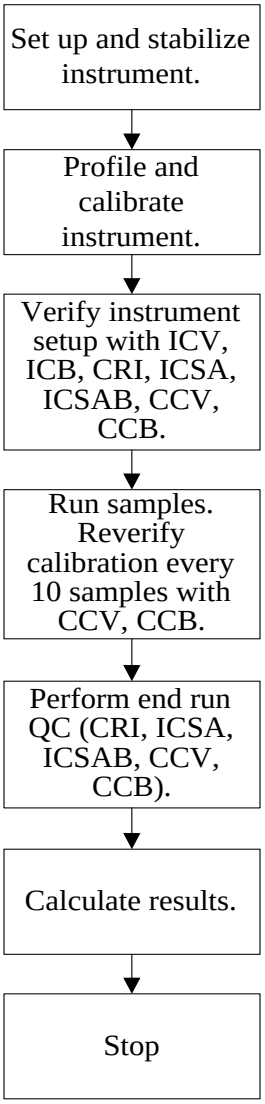
18.1.4. Added to section 8: Dissolved metals samples that are filtered and preserved at the laboratory with concentrated Nitric acid will be held for 24 hours before digestion.

18.2. Revision 11:

18.2.1. Added to section 9.9: For method 6010C the RLV/CRA which is the low level quantitation check sample is prepared and analyzed quarterly or as needed. The control limit is 70-130%. Please note the RLV/CRA (undigested) is still analyzed at the beginning and end of the analytical run.

18.2.2. Added references to SOP PT-QA-029.

Figure 1. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7, 6010B and 6010C Target Analyte List

ELEMENT	Symbol	CAS #	6010B/6010 C analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil	Reporting Limit (ug/wipe) Wipe
Aluminum	Al	7429-90-5	X	X	200	20	10
Antimony	Sb	7440-36-0	X	X	60	6	3
Arsenic	As	7440-38-2	X	X	300	30	15
Barium	Ba	7440-39-3	X	X	200	20	10
Beryllium	Be	7440-41-7	X	X	4	0.4	0.25
Boron	B	7440-42-8	X	X	200	20	10
Cadmium	Cd	7440-43-9	X	X	5	0.5	0.25
Calcium	Ca	7440-70-2	X	X	5000	500	250
Chromium	Cr	7440-47-3	X	X	10	1	0.5
Cobalt	Co	7440-48-4	X	X	50	5	2.5
Copper	Cu	7440-50-8	X	X	25	2.5	1.25
Iron	Fe	7439-89-6	X	X	100	10	5
Lead	Pb	7439-92-1	X	X	100	10	5
Lithium	Li	7439-93-2	X	X	50	5	2.5
Magnesium	Mg	7439-95-4	X	X	5000	500	250
Manganese	Mn	7439-96-5	X	X	15	1.5	0.75
Molybdenum	Mo	7439-98-7	X	X	40	4	2
Nickel	Ni	7440-02-0	X	X	40	4	2
Phosphorus	P	7723-14-0	X	X	300	30	NA
Potassium	K	9/7/7440	X	X	5000	500	250
Selenium	Se	7782-49-2	X	X	250	25	12.5
Silicon	Si	7631-86-9	X	X	500	N/A	N/A
Silver	Ag	7440-22-4	X	X	10	1	0.5
Sodium	Na	7440-23-5	X	X	5000	500	250
Strontium	Sr	7440-24-6	X	X	50	5	2.5
Thallium	Tl	7440-28-0	X	X	2000	200	100
Vanadium	V	7440-62-2	X	X	50	5	2.5
Zinc	Zn	7440-66-6	X	X	20	2	1

Note: Where reporting “Hardness” by ICP use the following equations per SM20th ed. 2340B:

Calcium Hardness = 2.497 [Ca, mg/L]
Total Hardness = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]
Where reporting "Silica" by ICP use the following equation:

Silica = Silicon * 2.14

TABLE IA. Method 200.7, 6010B and 6010C Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil	Reporting Limit (ug/wipe) Wipe
Arsenic	As	7440-38-2	10	1.0	0.5
Lead	Pb	7439-92-1	3.0	0.3	0.15
Selenium	Se	7782-49-2	5.0	0.5	0.25
Thallium	Tl	7440-28-0	10	1.0	0.5
Antimony	Sb	7440-36-0	10	1.0	0.5
Cadmium	Cd	7440-43-9	5.0	0.5	0.25
Silver	Ag	7440-22-4	5.0	0.5	0.25
Chromium	Cr	7440-47-3	5.0	0.5	0.25

TABLE II. Non-Routine Analyte List

			Reporting	Reporting	Reporting

ELEMENT	Symbol	CAS #	Limit (ug/L) Water	Limit (mg/kg) Soil	Limit (ug/wipe) Wipe
Tin	Sn	7440-31-5	100	10	5
Titanium	Ti	7440-32-6	50	5	2.5

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
Lithium	1000	1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	25000	50000
Antimony	10000	60	1000	5000
Arsenic	10000	300	1000	5000
Barium	10000	200	1000	5000
Beryllium	10000	4	1000	5000
Cadmium	10000	5	1000	5000
Calcium	100000	5000	25000	50000
Chromium	10000	10	1000	5000
Cobalt	10000	50	1000	5000
Copper	10000	25	1000	5000
Iron	100000	100	25000	50000
Lead	10000	100	1000	5000
Lithium	10000	50	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Strontium	10000	50	1000	5000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Boron	10000	200	1000	5000
Silicon	10000	500	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

TABLE IVA. 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	4	1000	2000
Cadmium	1000	5	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	250000	5000	50000	125000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	250000	5000	50000	125000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000
Boron	4000	200	1000	2000

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Silicon	4000	500	1000	2000
Tin	4000	100	1000	2000
Titanium	4000	50	1000	2000

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

* Custom TestAmerica solutions contain common analytes. 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	RSD between duplicate exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010B & 6010C: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate.
RLV/CRA/LLICV/LLCCV	Beginning of every analytical run, immediately following the ICB. Method 6010C also requires analysis at the end of the analytical sequence.	50 – 150% recovery. (1) Method 6010C: 70 – 130% recovery	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7, 6010B & 6010C: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.

TABLE VII. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICSA	Beginning of every run	See Section 9.10.3 [(1)]	See Section 9.10.3.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.10.2.
Dilution Test	One per prep batch.	For samples > 50x MDL, 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. [(1)] Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.3). Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.3).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.3 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. (85-115% for 200.7)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

TABLE VII. Summary of Quality Control Requirements			
QC PARAMETER	**FREQUENCY**	**ACCEPTANCE CRITERIA**	**CORRECTIVE ACTION**
		Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.7) may not require redigestion or reanalysis (see Section 9.4)	
Matrix Spike	One per sample preparation batch of up to 20 samples (6010B/6010C) or one per every 10 or fewer samples (200.7).	75 - 125 % (6010B & 6010C) or 70 – 130% (200.7) recovery or in-house control limits. (1) For TCLP See Section 10.15.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 10.15.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20%. (1)	See Corrective Action for Matrix Spike.

(1) For specific DoD requirements, refer to PT-QA-025. For DoD V4.1 refer to SOP PT-QA-029.

APPENDIX B

TESTAMERICA ICP DATA REVIEW CHECKLIST

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Pittsburgh ICP Data Review Checklist

Run Date: _____ 3. _____ 10. _____ 17. _____ 24. _____ 31. _____
Analyst: _____ 4. _____ 11. _____ 18. _____ 25. _____ 32. _____
Inst: _____ 6. _____ 12. _____ 19. _____ 26. _____ 33. _____
Meth: _____ 8. _____ 13. _____ 20. _____ 27. _____ 34. _____
Lots Analyzed: 7. _____ 14. _____ 21. _____ 28. _____ 35. _____
1. _____ 8. _____ 15. _____ 22. _____ 29. _____ 36. _____
2. _____ 9. _____ 16. _____ 23. _____ 30. _____ 37. _____

Review Item	Yes (✓)	No (✓)	N/A (✓)	2nd Lv (✓)	Comments
A. Calibration/Instrument Run QC					
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels?					
2. ICV/CCV analyzed at appropriate frequency and within control limits? (6010B, CLP=90-110%, 200.7=95-105%[ICV])?					
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?					
4. CRA/RLV/CRI analyzed? (CRI for CLP only)					
5. ICSA/ICSAB run at required frequency and within SOP limits?					
B. Sample Results					
1. Were samples with concentrations > the linear range for any parameter 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?					
5. Dilution Test done per prep batch (or per SDG for CLP)?					
6. Post digestion spike analyzed if required (CLP only)?					
D. Other					
1. Are all nonconformances documented appropriately?					
2. Current IDL/LR/IEC data on file?					
3. Calculations checked for error?					
4. Transcriptions checked for error?					
5. All client/project specific requirements met?					
6. Date/Time of analysis verified as correct?					

General Comments: _____

Analyst & Date: _____ Second-Level Review & Date: _____

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 6010C, 200.7 AND BY TESTAMERICA

CROSS REFERENCE OF TERMS COMMONLY USED IN

METHODS EPA 200.7, SW846 6010B/6010C AND TESTAMERICA INC. SOP

EPA 200.7	SW6010B/6010C	TestAmerica Inc. 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)

EPA 200.7	SW6010B/6010C	TestAmerica Inc. SOP
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 D

MSA GUIDANCE

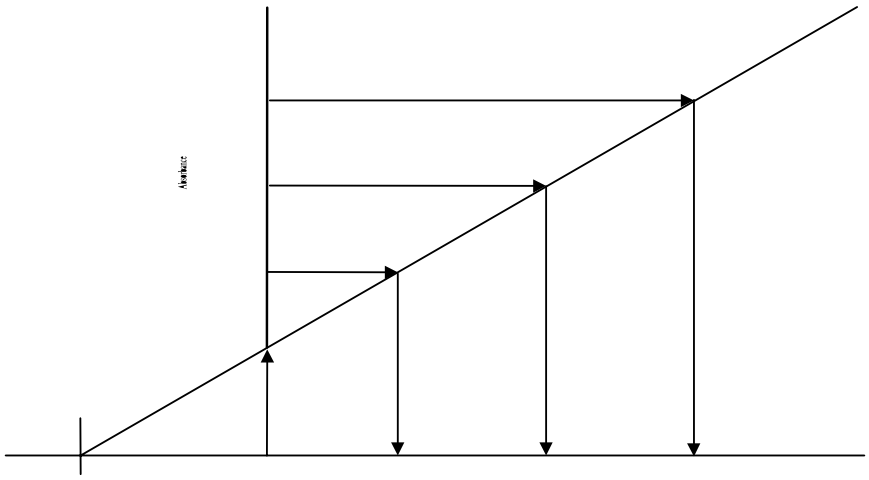
Appendix D. MSA Guidance

Controlled Source: Intranet

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 E

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
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged)

Problem	Possible Cause/Solution
	Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. 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 20% 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 pipette 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 G

PREVENTATIVE MAINTENANCE

APPENDIX G. 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 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 droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check Cu/Mn ratio-should be 30% of value at date that IECs were performed 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
Weekly	Apply silicon spray on autosampler tracks Check water level in cool flow
Monthly	Clean air filters on back of power unit to remove dust Check D mirror for air instruments
Bi-yearly	Change oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

Appendix D

Air Toxics, Ltd. Documentation

CERTIFICATE OF ACCREDITATION

ANSI-ASQ National Accreditation Board/ACLASS
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

Air Toxics, Ltd.
180 Blue Ravine Rd. Suite B
Folsom, CA 95630

has been assessed by ACLASS
and meets the requirements of

DoD-ELAP

while demonstrating technical competence in the field(s) of

TESTING

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

ADE-1451
Certificate Number

ACLASS Approval

Certificate Valid: 04/27/2010-04/27/2012
Version No. 001

SCOPE OF DoD-ELAP ACCREDITATION

Air Toxics, Ltd.

180 Blue Ravine Rd. Suite B, Folsom, CA 95630
Melanie Levesque Phone: 916-985-1000

TESTING

Valid to: April 27, 2012 Certificate Number: ADE- 1451

I. Environmental

MATRIX	SPECIFIC TEST or GROUP of ANALYTES	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Air and Emissions	BTEX / TPH	Modified EPA TO-3	GC/PID/FID
Air and Emissions	VOCs	Modified EPA TO-14A	GC/MS
Air and Emissions	VOCs	Modified EPA TO-15	GC/MS
Air and Emissions	SVOCs and VOCs	Modified EPA TO-17	GC/MS
Air and Emissions	Natural Gases and NMOC	Modified ASTM D-1945	GC/FID/dual TCD

Notes:

1. ** = As Applicable*
2. *This scope is part of and must be included with the Certificate of Accreditation No. ADE- 1451*

Vice President

DoD ELAP -- PT Performance Summary Review				Air ONLY	
Lab Name :	Air Toxics				
City/State :	Folsom, CA				
PT Provider Used :	ERA				
PartName	PartNumber	NELACCode	AnalyteName	EPAmethod#	Analyte Approved
Volatiles	PT-VOA-AIR	4375	Benzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4395	Bromodichloromethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4400	Bromoform	TO-15	Approved
Volatiles	PT-VOA-AIR	4950	Bromomethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4410	2-Butanone (MEK)	TO-15	Approved
Volatiles	PT-VOA-AIR	5000	tert-Butyl methyl ether (MTBE)	TO-15	Approved
Volatiles	PT-VOA-AIR	4455	Carbon tetrachloride	TO-15	Approved
Volatiles	PT-VOA-AIR	4475	Chlorobenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4575	Chlorodibromomethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4485	Chloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4505	Chloroform	TO-15	Approved
Volatiles	PT-VOA-AIR	4960	Chloromethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4555	Cyclohexane	TO-15	Approved
Volatiles	PT-VOA-AIR	4585	1,2-Dibromoethane (EDB)	TO-15	Approved
Volatiles	PT-VOA-AIR	4610	1,2-Dichlorobenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4620	1,4-Dichlorobenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4625	Dichlorodifluoromethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4630	1,1-Dichloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4635	1,2-Dichloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4640	1,1-Dichloroethene	TO-15	Approved
Volatiles	PT-VOA-AIR	4645	cis-1,2-Dichloroethene	TO-15	Approved
Volatiles	PT-VOA-AIR	4655	1,2-Dichloropropane	TO-15	Approved
Volatiles	PT-VOA-AIR	4680	cis-1,3-Dichloropropene	TO-15	Approved
Volatiles	PT-VOA-AIR	4685	trans-1,3-Dichloropropene	TO-15	Approved
Volatiles	PT-VOA-AIR	4695	1,2-Dichlorotetrafluoroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4765	Ethylbenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4768	p-Ethyltoluene	TO-15	Approved
Volatiles	PT-VOA-AIR	4825	n-Heptane	TO-15	Approved
Volatiles	PT-VOA-AIR	4855	n-Hexane	TO-15	Approved
Volatiles	PT-VOA-AIR	4860	2-Hexanone	TO-15	Approved
Volatiles	PT-VOA-AIR	4995	4-Methyl-2-pentanone (MIBK)	TO-15	Approved
Volatiles	PT-VOA-AIR	5067	Propylene	TO-15	Approved
Volatiles	PT-VOA-AIR	5110	1,1,2,2-Tetrachloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5115	Tetrachloroethylene	TO-15	Approved
Volatiles	PT-VOA-AIR	5140	Toluene	TO-15	Approved
Volatiles	PT-VOA-AIR	5160	1,1,1-Trichloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5165	1,1,2-Trichloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5175	Trichlorofluoromethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5195	Trichlorotrifluoroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5210	1,2,4-Trimethylbenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	5215	1,3,5-Trimethylbenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	5235	Vinyl chloride	TO-15	Approved
Volatiles	PT-VOA-AIR	5260	Xylenes, total	TO-15	Approved

MSD-2 Standard TO 15 0.5 ppbv
4/9/10
Loaded 50 ml of # 1911-333
2.0 ppbv → 0.5 ppbv
Rtx-1 column

Report Date : 27-Apr-2010 15:35

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ID:	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08
FILENAME:	2040906	2040907	2040908	2040909	2040910	2040911	2040912	2040913
INJ.DATE:	09-APR-2010	09-APR-2010	09-APR-2010	09-APR-2010	09-APR-2010	09-APR-2010	09-APR-2010	09-APR-2010
INJ.TIME:	14:20	15:04	15:52	16:36	17:20	18:05	18:51	19:37

ppbv

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	RL (ppbv)	APH (ppbv)
1 Propylene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
2 Dichlorodifluoromethan	699.93	635.08	674.04	641.22	681.46	672.55	594.13	623.72	652.77	35.09	105.20	0.5	
3 Chloromethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
4 Freon 114	542.85	567.18	605.72	549.04	610.03	582.29	552.13	538.53	568.47	28.04	84.07	0.5	
5 Vinyl Bromide	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
6 Freon134a	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
7 Vinyl Chloride	591.41	528.64	581.69	502.15	563.35	512.45	508.50	441.41	528.70	49.19	147.47	0.5	
8 1,3-Butadiene	699.40	526.57	646.61	565.32	575.47	453.96	539.92	427.75	554.37	90.40	271.02	0.5	0.9
9 Butane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
10 Isobutylene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
11 Bromomethane	811.84	592.02	780.62	605.83	738.37	592.09	592.30	517.25	653.79	107.28	321.63	0.5	
12 Chloroethane	645.80	525.18	593.60	502.23	631.70	453.70	436.06	526.87	539.39	77.96	233.72	+	
13 Ethanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
14 Acetone	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
15 Isopentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
16 Trichlorofluoromethane	590.11	627.96	625.80	651.17	643.39	610.35	581.06	621.66	618.94	24.24	72.66	0.5	
17 2-Propanol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		

Reviewer 1 Diane Benton Date: 4/27/10
Reviewer 2 James Scott Date: 5/12/10

$\bar{x}$ = 0.11
$\bar{x}$ = 0.108 5/12/10

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ppbv RL (ppbv) APH (ppbv)

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	
	18 Freon 22	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	19 Freon 152A	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	20 Acrolein	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	21 2,4-Dimethylpentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	22 tert-butyl alcohol	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	23 1,1-Dichloroethene	394.44	343.49	392.78	342.00	365.02	421.90	313.90	340.99	364.32	35.96	107.81	0.5
	24 Methylene Chloride	541.14	498.70	501.71	441.06	471.78	497.46	450.03	415.91	477.22	40.34	120.94	↓
	25 3-Chloroprene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	26 Methyl Acetate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	27 Freon 113	455.98	479.46	476.31	456.73	477.30	527.97	430.00	469.80	471.69	28.01	83.98	0.5
	28 Carbon Disulfide	515.86	478.35	502.31	452.26	501.83	454.53	440.30	439.79	473.16	30.47	91.35	↓
	29 Acetonitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	30 Acrylonitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	31 trans-1,2-Dichloroethe	525.11	458.96	440.44	348.76	489.88	390.32	295.47	397.09	418.25	75.43	226.14	0.5
	32 1,1-Dichloroethane	464.41	504.71	496.92	457.11	467.31	495.76	462.32	479.64	478.52	18.40	55.15	↓ JR 5/12/10
	33 MTBE	408.84	428.46	367.51	396.60	383.78	376.28	376.13	367.00	388.08	21.72	65.12	0.6
	34 Vinyl Acetate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	35 2-Butanone	424.99	409.95	380.41	339.30	364.59	388.30	363.28	256.35	365.89	51.91	155.62	0.5
	36 cis-1,2-Dichloroethene	437.80	400.82	435.11	413.69	432.83	326.80	345.70	378.40	396.39	42.42	127.18	0.5
	37 Chlorprene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
*	38 Bromochloromethane	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	0.00	0.00	
	39 Pentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ppbv RL (ppbv) APH (ppbv)

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	
	40 Isopropyl ether	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	41 Hexane	356.16	358.46	338.75	335.20	330.47	281.02	318.59	304.62	327.91	26.01	77.99	0.5
	42 Chloroform	541.62	509.32	540.45	529.69	536.95	520.59	513.31	549.49	530.18	14.48	43.40	↓
	43 Tetrahydrofuran	459.33	540.35	362.46	414.24	432.41	390.64	431.51	417.90	431.10	52.84	158.41	
	44 1,1-Dicholoropropene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	45 Ethyl-tert-butyl ether	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
$	46 1,2-Dichloroethane-d4	28658.34	30149.12	29558.41	29378.38	29148.26	30656.96	29504.16	30768.68	29727.79	737.24	2210.26	
	47 1,2-Dichloroethane	673.77	699.11	625.73	650.42	688.27	661.31	638.31	636.10	659.13	26.24	78.68	0.5
	48 Ethyl Acetate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	49 1,1,1-Trichloroethane	518.27	555.13	516.12	516.32	523.78	532.47	502.42	505.30	521.23	16.68	50.02	0.5
	50 Thiophene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	51 Benzene	496.57	501.51	533.11	472.05	507.16	508.72	487.97	469.36	497.06	20.82	62.41	0.5
	52 Carbon Tetrachloride	531.99	547.27	542.84	557.10	530.99	599.20	515.88	503.62	541.11	29.03	87.03	↓
	53 Cyclohexane	367.42	363.55	335.11	333.21	323.17	346.23	324.74	352.92	343.29	16.95	50.82	
*	54 1,4-Difluorobenzene	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	0.00	0.00	
	55 2,3-Dimethylpentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	56 2-Methylpentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	57 tert-amyl methyl ether	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	58 1,2-Dichloropropane	578.60	544.74	579.07	561.11	553.38	569.87	563.55	522.30	559.08	18.95	56.80	0.5
	59 Bromodichloromethane	630.47	560.91	592.37	593.48	643.67	581.44	615.21	552.45	596.25	32.05	96.08	↓
	60 1,4-Dioxane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	61 Trichloroethene	555.92	524.70	506.78	505.89	505.07	518.93	534.23	463.76	514.41	26.80	80.34	0.5
	62 2,2,4-Trimethylpentane	360.02	348.23	361.32	373.50	332.38	354.50	327.83	301.63	344.93	23.11	69.30	↓
	63 2-Chloroethyl Vinyl Et	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

0.6

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ppbv

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	RL (ppbv)	APH (ppbv)
	64 Heptane	400.75	348.36	368.83	367.36	385.56	347.54	349.52	371.02	367.37	19.03	57.04	0.5	
	65 cis-1,3-Dichloropropen	509.43	432.67	464.15	392.18	433.93	433.42	433.58	394.23	436.70	37.56	112.59	↓	
	66 4-Methyl-2-pentanone	540.37	499.48	540.51	455.34	569.24	471.79	456.41	502.97	504.51	42.43	127.20		
	67 Methylcyclohexane	333.07	293.05	332.15	292.74	314.57	311.96	264.61	264.77	300.87	26.92	80.70		
	68 trans-1,3-Dichloroprop	442.76	395.93	452.11	393.44	421.46	376.16	351.19	350.91	398.00	38.53	115.51		
	69 1,1,2-Trichloroethane	510.47	576.73	527.23	525.54	599.54	538.30	506.05	439.61	527.93	48.12	144.26		
$	70 Toluene-d8	26254.88	26284.85	26664.53	26369.28	26185.53	25773.42	26597.73	26531.98	26332.77	283.90	851.14		
	71 Toluene	503.52	482.14	494.93	470.92	483.23	465.15	445.57	441.94	473.43	21.98	65.90	0.5	0.5
	72 2-Hexanone	347.80	353.34	368.30	346.52	370.00	313.57	321.82	329.04	343.80	20.76	62.23	↓	
	73 Dibromochloromethane	527.12	437.79	549.84	464.92	519.03	467.66	504.52	419.07	486.24	45.96	137.78		
	74 1,2-Dibromoethane	515.90	508.66	532.63	494.94	536.19	476.96	472.21	467.51	500.63	26.95	80.79		
	75 1,3-Dichloropropane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
	76 Octane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
	77 Tetrachloroethene	493.27	497.21	560.14	485.23	503.52	494.40	472.47	437.44	492.96	34.24	102.65	0.5	
	78 Dibromomethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
*	79 Chlorobenzene-d5	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	0.00	0.00		
	80 Indan	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
	81 1,1,1,2-Tetrachloroeth	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
	82 Chlorobenzene	533.78	519.12	521.92	555.36	570.94	491.46	496.63	486.39	521.95	30.47	91.36	0.5	
	83 Ethyl Benzene	379.89	382.33	402.23	374.12	371.47	331.29	387.55	318.77	368.46	28.59	85.70	↓	0.4
	84 2,3-Dimethylheptane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
	85 m,p-Xylene	371.60	366.03	379.73	341.77	355.87	307.01	311.68	303.51	342.15	30.93	92.71	0.5	0.4
	86 Bromoform	502.65	416.03	515.82	413.13	502.20	432.68	460.27	386.68	453.68	48.77	146.21	↓	

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

pptv

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	RL (ppbv)	APH (ppbv)
87	Styrene	346.10	297.32	345.55	273.98	321.13	287.02	292.68	271.04	304.35	29.86	89.51	0.5	
88	Indene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
89	1,1,2,2-Tetrachloroeth	571.21	503.25	578.45	524.93	597.75	552.16	510.69	491.21	541.21	39.17	117.44	0.5	
90	o-Xylene	391.86	327.20	372.43	312.27	343.92	306.15	294.50	262.12	326.30	42.21	126.54	↓	0.4
91	cis-1,4-dichloro-2-but	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
92	Nonane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
$ 93	Bromofluorobenzene	23879.17	24318.65	24556.77	24498.22	24712.45	24451.32	25626.79	25987.19	24753.82	700.51	2100.14		
94	alpha-pinene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
95	Cumene	437.92	399.11	399.76	396.07	386.37	373.29	340.24	333.77	383.32	33.99	101.89	0.5	
96	1,2,3-Trichloropropane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
97	trans-1,4-dichloro-2-b	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
98	Propylbenzene	381.78	339.12	359.71	329.02	365.75	327.65	311.51	303.70	339.78	27.25	81.71	0.5	
99	2-Chlorotoluene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
100	4-Chlorotoluene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
101	1-Methyl-3-ethyltoluen	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
102	4-Ethyltoluene	357.86	303.96	343.99	323.59	346.79	311.10	287.20	270.02	318.06	30.75	92.20	0.5	
103	1,3,5-Trimethylbenzene	348.79	287.38	324.82	283.96	310.37	271.80	270.39	250.15	293.46	32.37	97.05	↓	
104	tert-Butylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
105	1,2,4-Trimethylbenzene	339.13	288.92	321.06	299.20	313.29	272.12	250.46	266.46	293.83	30.06	90.13	0.5	
106	Decane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
107	sec-Butylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++		
108	alpha-chlorotoluene	296.41	232.08	297.23	237.58	290.61	253.65	250.12	192.53	256.28	36.86	110.50	0.5	
109	1,3-Dichlorobenzene	532.12	489.07	505.60	454.53	496.01	460.68	436.57	416.72	473.91	38.41	115.15	↓	
110	1,4-Dichlorobenzene	498.71	459.55	486.14	426.76	481.58	427.99	390.11	392.64	445.43	42.26	126.70		

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ppbv RL (ppbv) APH (ppbv)

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	
111 Butylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
112 p-Cymene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
113 1,2,3-trimethylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
114 1,2-Dichlorobenzene	498.09	458.13	475.33	456.30	473.83	455.03	434.08	408.28	457.38	27.28	81.80	0.5
115 Butylcyclohexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
116 Undecane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
117 Hexachloroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
118 1,3,5-Trichlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
119 1,2-dibromo-3-chloropr	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
120 1,2,4-Trichlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
121 Dodecane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
122 Naphthalene	378.83	311.39	316.88	287.20	316.61	276.14	279.49	249.40	301.99	38.89	116.60	0.5 0.3
123 Hexachlorobutadiene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
124 1,2,3-trichlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

msd-2 Standard TO15 2.0ppbv
4/9/10

Loaded 200ml of #911-333
2.0ppbv → 2.0ppbv
Rtx-1 Column

Report Date : 27-Apr-2010 16:03

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ID:	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08
FILENAME:	2040914	2040915	2040916	2040917	2040918	2040919	2040920	2040921
INJ.DATE:	09-APR-2010	09-APR-2010	09-APR-2010	09-APR-2010	09-APR-2010	09-APR-2010	10-APR-2010	10-APR-2010
INJ.TIME:	20:20	21:00	21:39	22:18	22:57	23:36	00:15	00:55

pptv

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	RL (ppbv)
1 Propylene	3248.63	2745.36	2893.80	3079.08	3087.88	2456.32	3413.24	2804.13	2966.06	304.12	911.75	2.0
2 Dichlorodifluoromethan	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
3 Chloromethane	2157.19	2184.64	2052.13	1992.00	2038.62	1930.18	1991.63	1982.99	2041.17	88.44	265.13	2.0
4 Freon 114	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
5 Vinyl Bromide	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
6 Freon134a	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
7 Vinyl Chloride	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
8 1,3-Butadiene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
9 Butane	2398.41	1485.07	1646.49	1676.25	1920.08	1804.23	1871.64	1634.28	1804.56	278.52	835.01	2.0
10 Isobutylene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
11 Bromomethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
12 Chloroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
13 Ethanol	2650.08	2014.54	2055.76	2070.84	1805.62	2066.70	1834.34	1881.66	2047.44	266.36	798.53	2.0
14 Acetone	3175.28	3323.62	2912.25	2965.16	3078.39	3185.01	3334.81	2643.64	3077.27	231.78	694.88	↓
15 Isopentane	1585.25	1677.47	1687.40	1661.11	1695.65	1593.74	1631.27	1635.57	1632.18	57.77	173.20	5/12/10
16 Trichlorofluoromethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
17 2-Propanol	1734.97	1650.71	1591.80	1665.87	1697.92	1592.15	1542.27	1588.94	1633.08	64.97	194.77	2.0

Reviewer 1 Rhonda Benton Date: 4/27/10
Reviewer 2 _____ Date: _____

$\bar{X} = 0.36$

Report Date : 27-Apr-2010 16:03

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

pptv

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	RL (ppbv)
18 Freon 22	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
19 Freon 152A	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
20 Acrolein	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
21 2,4-Dimethylpentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
22 tert-butyl alcohol	1695.25	1714.15	1662.55	1721.43	1687.50	1676.20	1684.58	1668.78	1688.80	20.76	62.24	2.0
23 1,1-Dichloroethene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
24 Methylene Chloride	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
25 3-Chloroprene	1679.55	1389.67	1411.85	1390.40	1483.87	1413.60	1389.06	1449.85	1450.98	98.21	294.44	2.0
26 Methyl Acetate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
27 Freon 113	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
28 Carbon Disulfide	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
29 Acetonitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
30 Acrylonitrile	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
31 trans-1,2-Dichloroethe	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
32 1,1-Dichloroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
33 MTBE	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
34 Vinyl Acetate	1498.39	1439.96	1538.98	1492.50	1587.05	1516.73	1631.51	1537.80	1530.37	59.03	176.96	2.0
35 2-Butanone	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
36 cis-1,2-Dichloroethene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
37 Chlorprene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
* 38 Bromochloromethane	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	0.00	0.00	
39 Pentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ppbv RL (ppbv)

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	
	40 Isopropyl ether	1404.18	1445.72	1463.18	1292.50	1416.65	1289.19	1411.71	1330.98	1381.76	68.09	204.13	2.0
	41 Hexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	42 Chloroform	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	43 Tetrahydrofuran	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	44 1,1-Dicholoropropene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	45 Ethyl-tert-butyl ether	1385.14	1312.18	1349.92	1322.41	1465.27	1285.98	1315.52	1281.98	1339.80	60.71	181.99	2.0
$	46 1,2-Dichloroethane-d4	29698.98	29813.50	29173.83	30647.61	29920.09	29974.60	29834.43	29831.13	29861.77	403.10	1208.51	
	47 1,2-Dichloroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	48 Ethyl Acetate	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	49 1,1,1-Trichloroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	50 Thiophene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	51 Benzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	52 Carbon Tetrachloride	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	53 Cyclohexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
*	54 1,4-Difluorobenzene	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	0.00	0.00	
	55 2,3-Dimethylpentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	56 2-Methylpentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	57 tert-amyl methyl ether	1687.32	1611.10	1697.02	1680.02	1676.73	1571.96	1562.51	1619.57	1638.28	53.86	161.46	2.0
	58 1,2-Dichloropropane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	59 Bromodichloromethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	60 1,4-Dioxane	1513.48	1509.30	1536.27	1544.84	1646.39	1526.33	1646.47	1631.72	1569.35	61.00	182.88	2.0
	61 Trichloroethene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	62 2,2,4-Trimethylpentane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
	63 2-Chloroethyl Vinyl Et	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ppbv RL (ppbv)

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	
64	Heptane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
65	cis-1,3-Dichloropropen	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
66	4-Methyl-2-pentanone	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
67	Methylcyclohexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
68	trans-1,3-Dichloroprop	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
69	1,1,2-Trichloroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
$ 70	Toluene-d8	26190.57	25949.48	26224.59	25763.68	26022.20	25727.66	26306.27	25674.74	25982.40	244.05	731.67	2.0
71	Toluene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
72	2-Hexanone	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
73	Dibromochloromethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
74	1,2-Dibromoethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
75	1,3-Dichloropropane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
76	Octane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
77	Tetrachloroethene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
78	Dibromomethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
* 79	Chlorobenzene-d5	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	25000.00	0.00	0.00	
80	Indan	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
81	1,1,1,2-Tetrachloroeth	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
82	Chlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
83	Ethyl Benzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
84	2,3-Dimethylheptane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
85	m,p-Xylene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
86	Bromoform	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
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Inst ID: msd2.i

	Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL
87	Styrene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
88	Indene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
89	1,1,2,2-Tetrachloroeth	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
90	o-Xylene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
91	cis-1,4-dichloro-2-but	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
92	Nonane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
$ 93	Bromofluorobenzene	26240.41	26560.84	25870.42	26703.91	26272.43	25894.45	25996.93	26953.58	26311.62	396.85	1189.76
94	alpha-pinene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
95	Cumene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
96	1,2,3-Trichloropropane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
97	trans-1,4-dichloro-2-b	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
98	Propylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
99	2-Chlorotoluene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
100	4-Chlorotoluene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
101	1-Methyl-3-ethyltoluen	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
102	4-Ethyltoluene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
103	1,3,5-Trimethylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
104	tert-Butylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
105	1,2,4-Trimethylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
106	Decane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
107	sec-Butylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
108	alpha-chlorotoluene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
109	1,3-Dichlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++
110	1,4-Dichlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++

Air Toxics Ltd.
METHOD DETECTION LIMIT SUMMARY REPORT

Method File: /chem/msd2.i/09apr10.b/210q0407a.m
Batch File: /chem/msd2.i/09apr10.b
Inst ID: msd2.i

ppbv RL (ppbv)

Compound	MDL01	MDL02	MDL03	MDL04	MDL05	MDL06	MDL07	MDL08	AVG CONC	STD DEV	MDL	
111 Butylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
112 p-Cymene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
113 1,2,3-trimethylbenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
114 1,2-Dichlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
115 Butylcyclohexane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
116 Undecane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
117 Hexachloroethane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
118 1,3,5-Trichlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
119 1,2-dibromo-3-chloropr	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
120 1,2,4-Trichlorobenzene	1918.53	1701.04	1780.33	1731.96	1845.97	1671.07	1704.32	1711.19	1758.05	84.99	254.79	2.0
121 Dodecane	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
122 Naphthalene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	
123 Hexachlorobutadiene	2267.54	2159.90	2288.29	2215.83	2507.24	2226.48	2350.24	2313.90	2291.18	105.80	317.18	2.0
124 1,2,3-trichlorobenzene	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	+++++	

7.0 TO-14A/TO-15 – VOLATILE ORGANIC COMPOUNDS

This method involves full scan GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds using EPA Method TO-14A/TO-15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and is either concentrated using a cryogenic trap and/or concentrated using a hydrophobic multisorbent bed. The hydrophobic multisorbent bed functions as a drying system which removes water from the sample stream prior to analysis by full scan GC/MS. During analysis, the sample may be focused onto a cryogenic cooled column and/or a cryogenic cooled sleeve for analysis by full scan GC/MS.

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 7-1. Summary of Method Modifications

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Sample Drying System	Nafion Drier.	Multisorbent.	Multisorbent.
Blank acceptance criteria	< 0.2 ppbv.	< RL.	< RL.
Blanks and standards **(applies to Low Level analysis only)**	Zero Air.	Zero air.	Nitrogen.
BFB absolute abundance criteria	Within 10% of that from the previous day.	Not mandated.	CCV internal standard area counts are compared to ICAL, corrective action for > 40 %D.
Method Detection Limit	Not Specified.	Follow 40CFR Pt.136 App. B.	The MDL met all relevant requirements in Method TO-15 (statistical MDL less than the LOQ). The concentration of the spiked replicate may have exceeded 10X the calculated MDL in some cases.

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Initial Calibration	≤ 30 % RSD.	≤ 30 % RSD with 2 compounds allowed out to ≤ 40 % RSD.	≤ 30 % RSD with 2 compounds allowed out to ≤ 40 % for **QUAD and 5&20** analysis and 4 compounds allowed out to ≤ 40 % for **Low Level** analysis.
Daily CCV	≤ 30% D.	≤ 30% D.	For **QUAD and 5&20** analysis: 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated. If more than two compounds from the standard list recover outside of 70-130%, corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%. For **Low Level** analysis the above applies except corrective action will be taken if more than four compounds from the standard list recover outside of 70-130%.
Sample collection media.	Summa canister.	Summa canister.	Methods TO-14A/TO-15 are validated for samples collected in specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of these methods and not recommended for ambient or indoor air samples. Associated results are considered qualified.

Table 7-2. Method TO-14A/TO-15 Analyte List

Analyte	RL (ppbv) TO-15/ LL/5&20	%RSD	Acceptance Criteria	
			LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,1,2-Trichloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,1-Dichloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,1-Dichloroethene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2,4-Trichlorobenzene	2.0/0.5/20	30%	70 - 130	≤ 25
1,2,4-Trimethylbenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2-Dibromoethane (EDB)	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2-Dichlorobenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2-Dichloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2-Dichloropropane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,3,5-Trimethylbenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,3-Dichlorobenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,4-Dichlorobenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Benzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Bromomethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
Carbon Tetrachloride	0.5/0.1/5.0	30%	70 - 130	≤ 25
Chlorobenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Chloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
Chloroform	0.5/0.1/5.0	30%	70 - 130	≤ 25
Chloromethane	2.0/0.1/20	30%	70 - 130	≤ 25
Chlorotoluene (Benzyl Chloride)	0.5/0.1/5.0	30%	70 - 130	≤ 25
cis-1,2-Dichloroethene	0.5/0.1/5.0	30%	70 - 130	≤ 25
cis-1,3-Dichloropropene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Dichloromethane	0.5/0.2/5.0	30%	70 - 130	≤ 25
Ethylbenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Freon 11 (Trichlorofluoromethane)	0.5/0.1/5.0	30%	70 - 130	≤ 25
Freon 113 (Trichlorotrifluoroethane)	0.5/0.1/5.0	30%	70 - 130	≤ 25
Freon 114	0.5/0.1/5.0	30%	70 - 130	≤ 25
Freon 12 (Dichlorodifluoromethane)	0.5/0.1/5.0	30%	70 - 130	≤ 25
Hexachlorobutadiene	2.0/0.5/20	30%	70 - 130	≤ 25
m,p-Xylene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Methyl Chloroform	0.5/0.1/5.0	30%	70 - 130	≤ 25
o-Xylene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Styrene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Tetrachloroethene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Toluene	0.5/0.1/5.0	30%	70 - 130	≤ 25
trans-1,3-Dichloropropene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Trichloroethene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Vinyl Chloride	0.5/0.1/5.0	30%	70 - 130	≤ 25

Table 7-3. Method TO-14A/TO-15 Analyte List

Analyte	RL (ppbv) TO-15/ LL/5&20	%RSD	Acceptance Criteria	
			LCS (%R)	Precision Limits
1,3-Butadiene	0.5/0.1/5.0	30%	60 – 140	≤ 25
1,4-Dioxane	2.0/0.1/20	30%	60 – 140	≤ 25
2-Butanone (Methyl Ethyl Ketone)	0.5/0.1/5.0	30%	60 – 140	≤ 25
2-Hexanone	2.0/0.5/20	30%	60 – 140	≤ 25
4-Ethyltoluene	0.5/0.1/5.0	30%	60 – 140	≤ 25
4-Methyl-2-Pentanone (MIBK)	0.5/0.1/20	30%	60 – 140	≤ 25
Acetone	2.0/0.5/20	30%	60 – 140	≤ 25
Bromodichloromethane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Bromoform	0.5/0.1/5.0	30%	60 – 140	≤ 25
Carbon Disulfide	0.5/0.5/5.0	30%	60 – 140	≤ 25
Cyclohexane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Dibromochloromethane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Ethanol	2.0/0.5/20	30%	60 – 140	≤ 25
Heptane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Hexane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Isopropanol	2.0/0.5/20	30%	60 – 140	≤ 25
Methyl t-Butyl Ether (MTBE)	0.5/0.1/5.0	30%	60 – 140	≤ 25
Propylene	2.0/0.5/20	30%	60 – 140	≤ 25
Tetrahydrofuran	0.5/0.5/5.0	30%	60 – 140	≤ 25
trans-1,2-Dichloroethene	0.5/0.1/5.0	30%	60 – 140	≤ 25
2,2,4-Trimethylpentane	0.5/0.5/5.0	30%	60 – 140	≤ 25
Cumene	0.5/0.1/5.0	30%	60 – 140	≤ 25
Propylbenzene	0.5/0.1/5.0	30%	60 – 140	≤ 25
3-Chloroprene	2.0/0.5/20	30%	60 – 140	≤ 25
Naphthalene	2.0/0.5/20	30%	60 – 140	≤ 25
TPH (Gasoline) or NMOC (Hexane/Heptane)	10/2.0/50	One Point Calibration	NA	≤ 25

Table 7-4. Internal Standards

Analyte	Accuracy (% R)
Bromochloromethane	60 - 140
1,4-Difluorobenzene	60 - 140
Chlorobenzene-d_5	60 - 140

Table 7-5. Surrogates

Analyte	Accuracy (% R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

Table 7-6. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW – 846 tune criteria.	Correct problem then repeat tune.
5-Point Calibration	Prior to sample analysis.	% RSD ≤ 30 with two compounds allowed out to ≤ 40% RSD for QUAD and 5&20 (4 allowed out for LL).	Correct problem then repeat Initial Calibration Curve.
LCS	After each initial calibration curve, and daily, prior to sample analysis.	Recoveries for 90% of "Standard" compounds must be 70-130%; for 80% of "Non-standard" compounds, recoveries must be 60-140%. No recovery may be <50%. * If specified by the client in-house generated control limits may be used.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	For QUAD and 5&20: 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70-130%, corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%. For **Low Level** analysis the above applies except corrective action will be taken if more than four compounds from the standard list recover outside of 70-130%.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5 point calibration curve.
Laboratory	After the CCV/LCS.	Results less than the	Inspect the system and

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Blank		laboratory reporting limit.	Re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	**For blanks:** inspect the system and reanalyze the blank. **For samples:** re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70 - 130%. * If specified by the client in-house generated control limits may be used.	**For blanks:** inspect the system and reanalyze the blank. **For samples:** re-analyze the sample unless obvious matrix interference is documented. If the %R is within limits in the re-analysis, report the second analysis. If %R is out-of-limits a second time, then narrate results.
Laboratory Duplicates	10% of the samples.	RPD ≤25% for detections >5 X's the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found on the system, narrate results.