

*Final*

**Uniform Federal Policy – Quality Assurance Project Plan for  
Remedial Action**

**for the**

**Former U.S. Border Protection Firing Range**

**Nogales, AZ**

Prepared for:

U.S. Army Corps of Engineers, Fort Worth District

819 Taylor St., Room 3A12 Fort Worth, Texas 76102



Prepared by:

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Joint Venture (Sol-JCP, LLC)



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April 2019

UFP-QAPP Authorizing Signature:

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Steve Martin  
Contracting Officer's Representative  
US Army Corp of Engineers, Fort Worth District

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Date

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## ACRONYMS AND ABBREVIATIONS

ADEQ	Arizona Department of Environmental Quality
APP	Accident Prevention Plan
BGS	Below Ground Surface
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CO	Contracting Officer
COC	Contaminant of Concern
COR	Contracting Officer's Representative
DL	Detection Limit
DOD	Department of Defense
DQI	Data Quality Indicator
GC/MS	Gas Chromatography/Mass Spectrometry
GPL	Groundwater Protection Levels
HAZWOPER	Hazardous Waste Operations and Emergency Response
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	Interference check solutions
ICV	Initial Calibration Verification
IDQTF	Intergovernmental Data Quality Task Force
IDW	Investigation-Derived Waste
LCS	Laboratory Control Sample
LOD	Limit of Detection
LOQ	Limit of Quantitation
mg/kg	Milligram Per Kilogram
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
OSHA	Occupational Safety and Health Administration
PAH	Polycyclic aromatic hydrocarbons

PAL	Project Action Levels
PARCC	Precisions, Accuracy, Representativeness, Completeness, and Comparability
PM	Project Manager
PMP	Project Management Plan
POC	Point of Contact
PPE	Personal Protective Equipment
PQO	Product Quality Objectives
QA/QC	Quality Assurance/Quality Control
QCSR	Quality Control Summary Report
QL	Quantitation Limit
QSM	Quality System Manual
RACR	Remedial Action Completion Report
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOP	Standard Operating Procedures
SRL	Soil Remediation Level
SSHPP	Site Safety and Health Plan
TCLP	Toxicity Characteristics Leaching Procedure
UFP-QAPP	Uniform Federal Policy-Quality Assurance Project Plan
USACE	United States Army Corps of Engineer
USCBP	United States Customs and Border Patrol
USEPA	United States Environmental Protection Agency
XRF	X-ray Fluorescence

## Executive Summary

The Joint Venture formed by Sol Solutions, LLC and J.C. Palomar (Sol-JCP) has been retained by the United States Army Corps of Engineers (USACE) to provide a Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) Remedial Action for the removal of contaminated soil at the former U.S. Customs and Border Protection (USCBP) Firing Range (the Site). The former USCBP Firing Range (the Site) is located in Santa Cruz County in southern Arizona.

This Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP) was developed in conjunction with the Remedial Action Work Plan (RAWP) for the Site and presents the plan for collecting site-specific environmental data to support the remedial activities. This UFP-QAPP was prepared under Contract Number W9126G-17-C-0009, in accordance with the *UFP-QAPP* policy guidance (Intergovernmental Data Quality Task Force [IDQTF], 2012) to ensure the environmental data collected are scientifically sound, of known and documented quality, and suitable for the intended purposes.

This UFP-QAPP outlines the scope, field activities, and project-related activities associated with the remediation activities at the Site. As presented in the Decision Document (Terranear PMC, 2015) Alternative 4, Off-Site Disposal was selected as the remedial action for the Site.

The laboratory information cited in this UFP-QAPP is specific to Test America, located in Denver, Colorado. Test America – Denver was selected based on a competitive selection process among accredited laboratories. Test America – Denver has the necessary United States (U.S.) Department of Defense (DOD) Environmental Laboratory Accreditation Program (ELAP) certification required for this project, and is an Arizona certified laboratory. If additional laboratory services are requested that require modification to the existing UFP-QAPP, revised UFP-QAPP worksheets will be submitted to the USACE and regulatory agencies for approval.

This UFP-QAPP consists of the 37 worksheets specific to the UFP-QAPP guidance. All field standard operating procedures (SOPs) are provided in Appendix A. Laboratory SOPs are provided in Appendix B.

## Worksheet #1 – Title and Approval Page

**Project Name and Site** Former U.S Border Patrol Firing Range  
**Location:** Nogales, Arizona

**Document Title:** Former U.S Border Patrol Firing Range – Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP)

**Lead Organization:** U.S. Border Patrol

**Preparer’s Contact Information:** Sol Solutions, LLC and JC Palomar (Sol-JCP)  
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Scottsdale, AZ 85258  
Telephone: (916) 997-2842  
E-mail: [chris@jcpalomar.com](mailto:chris@jcpalomar.com)

**Preparation Date:** April 2019

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Chris Bason, Sol-JCP  
SOL-JCP Project Manager (PM)

Date

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Robert Noyes, Sol-JCP  
Sol-JCP Program Manager

Date

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David Clark, USACE  
USACE PM

Date

1 **Worksheet #2 – UFP-QAPP Identifying Information**

**Site Name/Number:** Former U.S Border Patrol Firing Range

**Contractor’s Name:** Sol Solutions, LLC / JC Palomar

**Contract Number:** W9126G-17-C-0009

**Contract Title:** Remedial Action at the Former U.S. Border Patrol Firing Range, Nogales, AZ

2 **1.** This UFP-QAPP was prepared in accordance with the requirements of the *UFP-QAPP* policy guidance  
 3 (IDQTF, 2012)

4 **2. Site Identification:**

Site ID	Site Name	Site Alias	Regulatory Program
Former U.S. Border Patrol Firing Range	Former U.S Border Patrol Firing Range	None	CERCLA

5  
 6 **3. Identify Regulatory Approval Entity:** Arizona Department of Environmental Quality (ADEQ), Region 7  
 7 United States Environmental Protection Agency (USEPA)

8 **4. This UFP-QAPP is a project-specific UFP-QAPP.**

9 **5. Dates of scoping sessions:**

Scoping Session	Date
Kick-off Meeting	March 15, 2017

10  
 11 **6. Dates and titles of any UFP-QAPP documents written for previous site work, if applicable:**

Title	Date
N/A	N/A

12  
 13 **7. Organizational partners (stakeholders) and connection with lead organization:**

Organization Partners/Stakeholders	Connection
U.S. Customs and Border Patrol (USCBP)	Lead Agency
ADEQ	Regulatory Oversight
USEPA	Regulatory Oversight
Sol Solutions, LLC / JC Palomar	Government Contractor

1 **8. List data users:** USCBP, ADEQ, USEPA, and Sol-JCP

2

3 **9. If any required UFP-QAPP elements are required and information are not applicable to the project,**  
4 **then circle the omitted UFP-QAPP elements and required information on the attached table. Provide**  
5 **an explanation for their exclusions below:** NOT APPLICABLE  
6

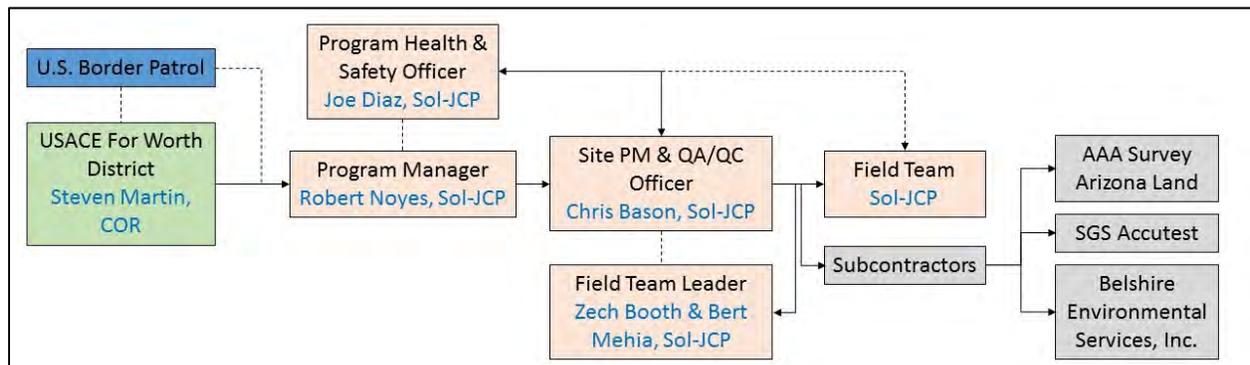
Required UFP-QAPP Element(s)	Required Information	Crosswalk to Related Documents
<b>Project Management and Objectives</b>		
Title and Approval Page	• Title and Approval Page	Worksheet #1
UFP-QAPP Identifying Information	• UFP-QAPP Identifying Information	Worksheet #2
Distribution List <ul style="list-style-type: none"><li>• Project Organization</li><li>• Distribution List</li></ul>	• Project Personnel List • Distribution List	Worksheet #3 Worksheet #5
Project Organization <ul style="list-style-type: none"><li>• Personnel Responsibilities and Qualifications</li><li>• Special Training Requirements and Certification</li><li>• Project Personnel Sign-Off Sheet</li></ul>	• Personnel Responsibilities and Qualifications Table • Special Personnel Training	Worksheet #4 Worksheet #7 Worksheet #8
Communication Pathways	• Communication Pathways	Worksheet #6
Project Planning/Problem Definition <ul style="list-style-type: none"><li>• Project Planning (Scoping)</li><li>• Problem Definition, Site History, and Background</li></ul>	• Project Scoping Session Participants Sheet • Problem Definition, Site History, and Background	Worksheet #9 Worksheet #10
Project Quality Objectives and Measurement Performance Criteria <ul style="list-style-type: none"><li>• Development of Project Quality Objectives Using the Systematic Planning Process</li><li>• Measurement Performance Criteria</li></ul>	• Site-specific Project Quality Objectives • Measurement Performance Criteria Table	Worksheet #11 Worksheet #12
<b>Project Management and Objectives</b>		
Secondary Data Evaluation	• Sources of Secondary Data and Information	Worksheet #13

Required UFP-QAPP Element(s)	Required Information	Crosswalk to Related Documents
	<ul style="list-style-type: none"> <li>• Secondary Data Criteria and Limitations Table</li> </ul>	
Project Overview and Schedule <ul style="list-style-type: none"> <li>• Project Overview</li> <li>• Project Schedule</li> </ul>	<ul style="list-style-type: none"> <li>• Summary of Project Tasks - Reference Limits and Evaluation Table</li> <li>• Project Schedule/Timeline Table</li> </ul>	Worksheet #14 Worksheet #15 Worksheet #16
<b>Measurement/Data Acquisition</b>		
Sampling Tasks <ul style="list-style-type: none"> <li>• Sampling Process Design and Rationale</li> <li>• Sampling Procedures and Requirements               <ul style="list-style-type: none"> <li>- Sampling Collection Procedures</li> <li>- Sample Containers, Volume, and Preservation</li> <li>- Equipment/Sample Containers Cleaning and Decontamination Procedures</li> <li>- Equipment Calibration, Maintenance, Testing, and Inspection Procedures</li> <li>- Supply Inspection and Acceptance Procedures</li> <li>- Field Documentation Procedures</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>• Sampling Design and Rationale</li> <li>• Sample Location Figures</li> <li>• Sampling Locations and Methods/SOP Requirements Table</li> <li>• Analytical Methods/SOP Requirements Table</li> <li>• Field Quality Control Sample Summary Table</li> <li>• Sampling standard operating procedures (SOPs) and</li> <li>• Project Sampling SOP References Table</li> <li>• Field Equipment Calibration, Maintenance, Testing, and Inspection Table</li> </ul>	Worksheet #17 Worksheet #18 Worksheet #19 Worksheet #20 Worksheet #21 Worksheet #30
Analytical Tasks <ul style="list-style-type: none"> <li>• Analytical SOPs</li> <li>• Analytical Instrument Calibration Procedures</li> </ul>	<ul style="list-style-type: none"> <li>• Analytical SOPs</li> <li>• Analytical SOP References Table</li> <li>• Analytical Instrument Calibration Table</li> </ul>	Worksheet #22 Worksheet #23 Worksheet #24 Worksheet #25

Required UFP-QAPP Element(s)	Required Information	Crosswalk to Related Documents
<ul style="list-style-type: none"> <li>Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures</li> <li>Analytical Supply Inspection and Acceptance Procedures</li> </ul>	<ul style="list-style-type: none"> <li>Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table</li> </ul>	
Sample Collection Documentation, Handling, Tracking, and Custody Procedures <ul style="list-style-type: none"> <li>Sample Collection Documentation</li> <li>Sample Handling and Tracking System</li> <li>Sample Custody</li> </ul>	<ul style="list-style-type: none"> <li>Sample Collection, Documentation Handling, Tracking, and Custody SOPs</li> </ul>	Worksheet #26 Worksheet #27
Quality Control Samples <ul style="list-style-type: none"> <li>Sampling Quality Control Samples</li> <li>Analytical Quality Control Samples</li> </ul>	<ul style="list-style-type: none"> <li>Quality Control Samples Table</li> </ul>	Worksheet #28
Data Management Tasks <ul style="list-style-type: none"> <li>Project Documentation and Records</li> <li>Data Package Deliverables</li> <li>Data Reporting Formats</li> <li>Data Handling and Management</li> <li>Data Tracking and Control</li> </ul>	<ul style="list-style-type: none"> <li>Project Documents and Records Table</li> <li>Analytical Services Table</li> <li>Data Management SOPs</li> </ul>	Worksheet #29
<b>Assessment/Oversight</b>		
Assessments and Response Actions <ul style="list-style-type: none"> <li>Planned Assessments</li> <li>Assessment Findings and Corrective Action Responses</li> <li>QA Reports</li> <li>Final SI Report</li> </ul>	<ul style="list-style-type: none"> <li>Assessments and Response Actions</li> <li>Planned Project Assessments Table</li> <li>Audit Checklists</li> <li>Assessment Findings</li> <li>QA Management Reports Table</li> </ul>	Worksheet #31 Worksheet #32 Worksheet #33
<b>Data Review</b>		
Data Review Steps <ul style="list-style-type: none"> <li>Step I: Verification</li> <li>Step II: Verification               <ul style="list-style-type: none"> <li>Step IIa Verification Activities</li> <li>Step IIb Verification Activities</li> </ul> </li> <li>Step III: Usability Assessment               <ul style="list-style-type: none"> <li>Data Limitations and Actions from Usability Assessment</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>Verification (Step I) Process Table</li> <li>Verification (Steps IIa and IIb) Process Table</li> <li>Verification (Steps IIa and IIb) Summary Table</li> <li>Usability Assessment</li> </ul>	Worksheet #34 Worksheet #35 Worksheet #36 Worksheet #37

## Worksheet #3 and #5 – Project Organization and UFP-QAPP Distribution List

Recipient	Title	Organization	Telephone Number	E-mail Address
David Clark	Project Manager (PM)	USACE	(817) 886-1876	<a href="mailto:David.S.Clark@usace.army.mil">David.S.Clark@usace.army.mil</a>
Steven Martin	Contracting Officer Representative (COR)	USACE	(817) 886-1873	<a href="mailto:Steven.G.Martin@usace.army.mil">Steven.G.Martin@usace.army.mil</a>
Joseph Zidron	USCBP PM	USCBP	(949) 643-6392	<a href="mailto:joseph.zidron@cbp.dhs.gov">joseph.zidron@cbp.dhs.gov</a>
Robert Noyes	Program Manager	Sol-JCP	(480) 544-7045	<a href="mailto:rnoyes@solsolutions.net">rnoyes@solsolutions.net</a>
Chris Bason	SOL-JCP Site PM, and Quality Assurance/Quality Control (QA/QC) Officer	Sol-JCP	(916) 997-2842	<a href="mailto:chris@jcpalomar.com">chris@jcpalomar.com</a>
Bert Mehia	Field Team Leader / Site Safety Coordinator	Sol-JCP	(714) 581-3005	<a href="mailto:bert@jcpalomar.com">bert@jcpalomar.com</a>
Nicole Osuch	Project Manager	ADEQ	(602) 771-4847	<a href="mailto:osuch.nichole@azdeq.gov">osuch.nichole@azdeq.gov</a>
TBD	Project Manager	USEPA	TBD	TBD
Joe Diaz	Program Health and Safety Officer	Sol-JCP		
Joy Chang	Client Relations Manager	Test America – Denver	(303) 467-7396	<a href="mailto:Joy.Chang@testamericainc.com">Joy.Chang@testamericainc.com</a>



*Project Organization Chart*

## Worksheet #4, #7, and #8 – Personnel Qualifications and Sign-off Sheet

This table lists the qualifications / training required for each project function. All training will occur prior to commencement of field activities. Training records will be stored onsite and in project files. This table will also serve as the signoff sheet to document that key project personnel are in possession of and have read the UFP-QAPP.

Name	Title/Role	Experience / Responsibilities	Specialized Training / Certifications	Signature/ Date
Robert Noyes	Sol-JCP Program Manager	26 years of experience. PM, primary point of contact (POC) for all project-related issues for the contract.	Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour Training, 30-hr Occupational Safety and Health Administration (OSHA) Site Supervisor Training	
Chris Bason	1. SOL-JCP Site PM 2. QA/QC Officer 3. Health & Safety Officer	24 years of experience. 1. Oversees project administration, monitors project performance, and directs and oversees project staff for the SOL-JCP Field Team. 2. Provides technical support and reviews technical deliverables. Oversees compliance with program and project-specific quality requirements 3. Responsible for health and safety performance, interacts with Site Safety Coordinator to ensure project-specific safety of field personnel.	HAZWOPER 40-hour Training, CPR, first aid, 30-hr OSHA Site Supervisor Training, OSHA Competent Person Training, USACE CQM Certified	
Zech Booth	SOL-JCP Field Team Leader / Site Safety Coordinator	16 years of experience. Perform or oversee the performance of activities required to complete remediation and sampling activities.	HAZWOPER 40-hour Training, CPR, first aid, 30-hour OSHA Site Supervisor Training, OSHA Competent Person Training	
Bert Mehia	SOL-JCP Field Team Leader / Site Safety Coordinator	12 years of experience. Perform or oversee the performance of activities required to complete remediation and sampling activities.	HAZWOPER 40-hour Training, 30-hour OSHA Site Supervisor Training, OSHA Competent Person Training	
Joe Diaz	Program Health and Safety Officer	30 years of experience. Responsible for health and safety performance. Interacts with Health and Safety Officer to ensure project-specific safety of field personnel.		
Diane Wisbeck	Program Chemist	25 years of experience.		

		Reviews UFP-QAPP and Quality Assurance (QA) input. Provides oversight of laboratory and reports data validation issues. Establishes the need for analytical data validations Corrective Actions.		
Joy Chang	PM/QAO	Responsible for laboratory QA program and review of QC data.		

## Worksheet #6 – Communication Pathways

Communication Driver	Organization	Name	Contact Information	Procedure (Timing, Pathway, Documentation, etc.)
Point of Contact (POC) for USACE – Contractual Issues and Deliverables	USACE COR	Steven Martin	(817) 886-1873 <a href="mailto:Steven.g.martin@usace.army.mil">Steven.g.martin@usace.army.mil</a>	Primary point of contact (POC) for USACE; can delegate communication to other internal or external points of contact. Will handle all contractual issues.
POC for USACE – Field Activities and Deliverables	USACE PM	David Clark	(817) 886-1876 <a href="mailto:David.s.clark@usace.army.mil">David.s.clark@usace.army.mil</a>	POC for USACE field work. Will approve any major changes to the UFP-QAPP before the changes can be implemented. During field work, if any sampling or sampling-related procedures in the UFP-QAPP need to be changed, the USACE PM officer will be notified 24 hours in advance of any changes to be made to the document.
Regulatory Oversight	ADEQ	Nicole Osuch	(602) 771-4847 <a href="mailto:osuch.nichole@azdeq.gov">osuch.nichole@azdeq.gov</a>	Primary POC for ADEQ; communicates directly (verbal and/or in writing) with USACE and Sol-JCP
Primary POC for Contractor, including providing program level communication and oversight of all field activities.	Sol-JCP Program Manager	Robert Noyes	(480) 544-7045 <a href="mailto:rnoyes@solsolutions.net">rnoyes@solsolutions.net</a>	Communicates directly (verbal and/or in writing) with USACE and Sol-JCP team members. To include invoicing related communication and preparation of monthly progress reports.
Communication with all field personnel, oversight of field activities, implementation of RAWP	SOL-JCP Site PM	Chris Bason	(916) 997-2842 <a href="mailto:chris@jcpalomar.com">chris@jcpalomar.com</a>	Communicates directly (verbal and/or in writing) with Sol-JCP PM on daily field activities. Monitors project performance, and directs and oversees project staff for the SOL-JCP Field Team. Provides technical support and reviews technical deliverables. Oversees compliance

				with program and project-specific quality requirements.
UFP-QAPP Changes in the Field; Field and Laboratory QA	Sol-JCP QA/QC Officer	Chris Bason	(916) 997-2842 <a href="mailto:chris@jcpalomar.com">chris@jcpalomar.com</a>	Communicates directly (verbal and/or in writing) with Sol-JCP PM on any UFP-QAPP changes in the field. Will serve as the primary POC for all field and laboratory QA issues. Responsible for confirming that the UFP-QAPP requirements are met by the field and laboratory staff. Provides direction regarding requirements for corrective actions for field and analytical issues.
Primary POC for Laboratory	Test America - Denver	Joy Chang	(303) 467-7396 <a href="mailto:Joy.Chang@testamericainc.com">Joy.Chang@testamericainc.com</a>	Primary POC for laboratory. Receives direction from Sol-JCP. Responsible for confirming the UFP-QAPP requirements are met by the laboratory.

## Worksheet #9 – Project Scoping Participants Sheet

A project kick-off meeting to discuss the Site Remediation Project occurred on March 15, 2017 (see the below participants' sheet). In attendance were representatives from the USCBP, USACE Fort Worth, and Sol-JCP. The main items of discussion were:

1. Roles/Responsibilities:
  - a. Chris Bason will be managing the field activities
  - b. Robert Noyes will be coordinating and submitting all reports/workplans (Project Management Plan [PMP], RAWP, UFP-QAPP, Accident Prevention Plan [APP], and Site Safety and Health Plan [SSHP])
2. Public Notice:
  - a. Location: Holiday Inn, Nogales
  - b. Steve Martin would be POC
  - c. Steve would follow-up with meeting time frames and newspaper to use
  - d. A public notice is required of the action followed by a public meeting before site mobilization in Nogales "no sooner than 30 days before site mob".
3. Site Access/Adjacent Properties:
  - a. Paul Argo is owner of leased property where former firing range is located
  - b. Adjacent property is owned by two brothers
  - c. Noel Garcia owns adjacent property
  - d. Water source/access would be provided by USCBP – location/access would be provided prior to start of field activities
4. Document Submittals:
  - a. Sol-JCP will provide Draft PMP
  - b. Sol-JCP will submit final documents to ADEQ
  - c. ADEQ POC is Nicole Osuch.
  - d. Monthly progress reports are required.
  - e. Pre-mobilization deliverables include PMP, UFP-QAPP, RAWP, APP/SSHP

Name	Affiliation	Title	Contact Information
Steve Martin	USACE Fort Worth	COR	817-886-1873 <a href="mailto:Steven.G.Martin@usace.army.mil">Steven.G.Martin@usace.army.mil</a>
Joseph Zidron	USCBP	USCBP PM	<a href="mailto:joseph.zidron@cbp.dhs.gov">joseph.zidron@cbp.dhs.gov</a>

David Clark	USACE Fort Worth	USACE Project Manager	(817) 886-1876 <a href="mailto:David.S.Clark@usace.army.mil">David.S.Clark@usace.army.mil</a>
Robert Noyes	Sol-JCP	Sol-JCP Program Manager	(480) 544-7045 <a href="mailto:rnoyes@solsolutions.net">rnoyes@solsolutions.net</a>
Chris Bason	Sol-JCP	SOL-JCP Site PM, and QA/QC Officer	(916) 997-2842 <a href="mailto:chris@jcpalomar.com">chris@jcpalomar.com</a>

## **Worksheet #10 – Problem Definition**

### **Scope**

The Joint Venture formed by Sol Solutions, LLC and J.C. Palomar (Sol-JCP) has been retained by the United States Army Corps of Engineers (USACE) to provide a Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) Remedial Action for the removal of contaminated soil at the former U.S. Customs and Border Protection (USCBP) Firing Range (the Site). This UFP-QAPP outlines the scope, field activities, and project-related activities associated with the remediation activities identified in the RAWP at the Site. As presented in the Decision Document (Terranear PMC, 2015) Alternative 4, Off-Site Disposal was selected as the remedial action for the Site.

### **Project Objectives**

The goals of the remedial action at the former USCBP Firing Range are:

- Effectively mitigate impacts to soil in a manner that provides short-term and long-term protection of human health and the environment.
- Conduct soil excavations to address soil contamination as identified in the 2014 RI Report (Terranear PMC, 2014).
- Prepare a Remedial Action Completion Report (RACR) summarizing the field activities and appropriate cleanup levels.

### **Site Location and Operational History**

The former USCBP Firing Range is located at 1651 W. Target Range Road in Nogales, Arizona (Figure 1). The area is defined as the leased portion of the Arbo property (parcel no. 112-29-010B) and a portion of the Barr property (parcel no. 113-49-027) (Figure 2). The site on the Arbo property (113-49-010B) is surrounded on two sides located in a portion of Section 13, Township 24 south, Range 13 east Santa Cruz County, Arizona with its center located at latitude of 31.347139 North and longitude of 110.969525 West.

The former USCBP Firing Range encompasses approximately one-half acre of shooting range property and empty range land used for small arms training in the form of target practice beginning in 1992. The USCBP discontinued use of the firing range in mid-2010. USCBP continues to lease the property from the current property owner, Mr. Arbo (Allwyn Environmental, 2009b). The property is currently idle.

The area within the former USCBP Firing Range currently consists of the firing range and firing range structures. An aerial photograph review conducted as part of Phase I ESAs (Allwyn Environmental, 2009a and b) of a property adjacent to the Site revealed that the structures present were constructed in 1992, and no previous development had occurred at the Site. The areas immediately surrounding the Site have never been developed. Operations ceased at the property in 2010 and it is no longer used as an active firing range.

The Site is located within a mostly open and moderately restricted area that is undeveloped property. The Site is unfenced, although there is a locked gate on the main road to the Site. There is no signage at the Site to indicate property boundaries or to ward off trespassers. It is possible for cattle and other livestock

from surrounding properties to enter the Site. At the completion of the Remedial Action, the USCBP will terminate the lease of this property and it will return to the property owner for use as agricultural land.

## **Previous Investigations**

The RI was completed at the Site in 2014 (Terranear PMC, 2014). A total of 60 soil samples were collected at the Site and analyzed for antimony, arsenic and lead (Figure 3) as follows:

- 38 soil samples (16 composite samples and 22 discrete samples) were collected from the surface (0-12 inches below ground surface [bgs]), and
- 22 soil samples (16 composite samples and 6 discrete samples) were collected between 12 and 42 inches bgs.

In addition, 10 of the surface soil samples were analyzed for polynuclear aromatic hydrocarbons (PAHs) and 5 samples were selected for Toxicity Characteristic Leaching Procedure (TCLP) analysis for antimony, arsenic, lead and PAHs.

Arsenic, antimony and lead were detected in all 60 samples. The highest concentration of metals and PAHs were found in the southwest corner of the firing range. This area comprises the major portion of the backstop berm and firing range area between the berm and last target area.

- **Antimony:** Antimony concentrations were detected at concentrations up to 454 milligrams per kilogram (mg/kg), with concentrations greater than the USEPA Regional Screening Level (RSL) for residential soil (31 mg/kg) in 27 of the 60 soil samples. The highest concentrations of antimony were detected in the soil samples BPN-13S (composite, west part of the firing range on the east slope of backstop berm) and BPG-3S (discrete 'grab', southwest part of the firing range on the east slope of backstop berm) both at 454 mg/kg. Therefore antimony was retained as a contaminant of concern (COC).
- **Arsenic:** Arsenic concentrations ranged from 4.4 mg/kg (composite sample BPN-14D14, central firing range) to 22.8 mg/kg (composite sample BPN-13S, west central firing range, east side of backstop berm) which are greater than the 2014 USEPA RSL for residential soil (0.39 mg/kg). However, these concentrations are consistent with both local and regional background levels of arsenic, ranging from 10 to 40 mg/kg (Shacklette and Boerngen, 1984). Therefore, arsenic was determined to not be a COC.
- **Lead:** Lead concentrations ranged from 198 mg/kg to 49,300 mg/kg in the shallow samples and from 20 mg/kg to 27,000 mg/kg in the deep samples. Of the 60 soil samples collected, 50 contained concentrations of lead above the USEPA RSL for residential soil (400 mg/kg). The highest concentration of lead (49,300 mg/kg) was detected in the discrete sample BPG-3S (southwest firing range, on eastern slope of backstop berm). Therefore lead was identified as the primary COC.
- **PAHs:** PAHs were detected in six of the nine shallow composite soil samples and in the one discrete shallow 'grab' soil sample (BPG-20S). Five composite soil samples and the discrete 'grab' soil sample contained concentrations exceeding their respective USEPA RSLs for residential soil for at least one of the following PAH compounds: benzo(a)anthracene, benzo(a)pyrene, and benzo(b)fluoranthene. Therefore, these PAHs were retained as COCs.

- TCLP: Four of the five TCLP samples exceeded the USEPA Regulatory Level for lead (5 milligrams per liter [mg/L]). Arsenic was detected in only one of the samples at a concentration that is less than the USEPA Regulatory Level for arsenic (5 mg/L). There are no USEPA Regulatory Levels for PAHs and antimony. The results of TCLP analyses of soil samples indicate that lead may be present at characteristically hazardous levels as defined by 40 CFR 261, Appendix II, 1993 ed., as amended by 58 FR 46040, August 31, 1993.

In September 2018, a background arsenic study was completed at the Site (HDR, 2018). A total of 10 soil samples were collected from a depth interval of 1-1.5 ft bgs and analyzed for arsenic. Arsenic was detected at concentrations ranging from 5.7 mg/kg to 11 mg/kg. The report concluded that the 95% UCL for arsenic was 7.55 mg/kg or 6.63 mg/kg (with or without the potential 11 mg/kg outlier). The arsenic concentrations are within the naturally occurring range in the state of Arizona, with a 95% UCL less than the Arizona Soil Remediation Level (SRL) of 10 mg/kg.

## **Environmental Setting**

### *Topography*

The majority of the former USCBP Firing Range has been graded by heavy machinery and is essentially flat. The topography of the remainder of the Site and of the surrounding property is typical of dry desert lowlands present throughout the Basin and Range province of the western United States. The land surface is generally rugged and hilly. Several dry creek beds (arroyos) separate steep hills and ridges present throughout this area. The elevation ranges from approximately 3,960 to 4,130 feet above sea level.

### *Climate*

Nogales' climate is typically sunny and dry, with low relative humidity. Average monthly high temperatures recorded at the Nogales 6 N climate station from 1952 to 2010 range from a low of 64.3 degrees Fahrenheit (°F) in January to a high of 95.3°F in June. Average monthly low temperatures range from 27.3°F in January to 63.9°F in June during the same time period (Western Regional Climate Center, 2011).

Nogales' climate is classified as arid, which is defined by average annual precipitation less than half of evaporation and mean temperature of the coldest month above freezing (32 °F). The former USCBP Firing Range receives little rain or snow, averaging about 17.21 inches of precipitation per year. Most precipitation occurs during the summer monsoon season, typically from July through mid-September. The monthly average precipitation recorded at the Nogales 6 N climate station from 1952 to 2010 ranges from a low average of 0.22 inches for May to a high average of 4.38 inches for August. The summer monsoon season for regional precipitation is characterized by incidences of sudden, dramatic downpours of heavy rain within a short period of time.

### *Soils and Vegetation*

The soils in the Site are primarily shallow and rocky with un-weathered clasts of andesite and rhyolite tuffs, granites, and small areas of clay shales. The steeper slopes have numerous rock outcroppings and shallow loamy soils. Five soil associations dominate the area: Comoro-Pima, Continental-Sonoita, Caralampi-White House - Hathaway, Lampshire-Chiracahua-Graham, and Faraway-Rock Outcrop-

Barkerville. The first three are typically deep soils and sandy loams with varying amounts of gravel and clay, generally appearing in or along floodplains and streambeds. The latter two are typically shallow cobbled clay or sandy loams occurring in the upper elevations on foothills and mountains (Allwyn Environmental, 2009c). Soil pH ranges from slightly acidic (pH 6) to slightly alkaline (pH 8) (USDA, 1979).

Most of the ground surface is bare, with portions partially covered with vegetation. The vegetation that grows in these soils is representative of desert shrub land. Common vegetation includes several varieties of cacti, mesquite, creosote bush, ocotillo, acacia trees, desert willow, and yucca (National Park Service, 2011). Vegetation at the former USCBP Firing Range did not significantly hinder the Remedial Investigation (RI) field activities (Terranear PMC, 2014).

### *Hydrogeology*

The Site lies within the boundaries of the Santa Cruz Active Management Area. The Santa Cruz Active Management Area was designed to address groundwater overdraft in the area, as a result, water management in this area is intensive. Within the Santa Cruz Active Management Area, groundwater can be withdrawn legally only through a groundwater right or permit, unless groundwater is withdrawn from an exempt well (maximum capacity of 35 gallons per minute or less). Based on the information provided in a well driller report from a well located within proximity to the site (Arizona Department of Water Resources Well No.55-636229), the local groundwater is located approximately 135 feet below land surface in this well which is cased to 420 feet below land surface. No perched water appears to exist in the area as no intermittent clay layers were noted. Based on site topography, the groundwater flow near the subject property is likely to the north to northeast.

### *Prehistoric and Historic Cultural Resources*

There are no identified prehistoric or historic cultural resources within the immediate vicinity of the former USCBP Firing Range property.

## **Remedial Action**

The activities associated with the remedial activities can be summarized as follows:

- Pre-Construction Activities
  - Site Security
  - Mobilization and Site Preparation
  - Truck Ingress/Egress
  - Topographic Survey
  - Utility Location
  - Clearing of Work Area
  - Removal of Structures
  - Erosion and Sediment Control
- Soil Excavation and Temporary Stockpiling
  - Soil Excavation
  - Temporary Stockpiles
  - Confirmatory Soil Sampling

- Disposal and Site Restoration
  - Waste Characterization
  - Disposal
  - Decontamination Procedures
  - Site Restoration
  - Demobilization
- Project-related activities
  - Laboratory analytical tasks
  - Data management and storage
  - Data review
  - Reporting

## Worksheet #11 – Project Quality Objectives/Systematic Planning Process Statements

### 1. What types of data are needed?

Both analytical and field data for soil are required for this project. All analytical samples will be submitted to the offsite subcontracted laboratory for analysis, Test America – Denver. Field screening for lead using a X-ray Fluorescence meter (XRF) will be conducted onsite by the Project Team (used to refer to SOL-JCP and its subcontractors). The following list presents a summary of the data required. Worksheets #17 and #18 of this UFP-QAPP provide the sample locations, numbers, rationale, and methodology.

Analytical data:

- Soil – Confirmation Sampling
  - Lead, arsenic, and antimony by USEPA SW-846 Method 6010C
  - PAHs by USEPA SW-846 Method 8270D
- Soil – Waste Characterization (Investigation-Derived Waste [IDW])
  - Resource Conservation and Recovery Act (RCRA) Characteristics
  - Toxicity Characteristic Leaching Procedure (TCLP) Parameters Lead
- Aqueous – Waste Characterization (IDW)
  - Lead, arsenic, and antimony by USEPA SW-846 Method 6010C
  - PAHs by USEPA SW-846 Method 8270D
- Field data
  - Soil screening using a XRF meter.

### 2. What are the Project Action Levels (PALs)?

The goal of the remedial action at the former USCBP Firing Range to effectively mitigate impacts to soil in a manner that provides short-term and long-term protection of human health and the environment. The Project Action Levels (PALs) are based on the USEPA Regional Screening Levels (RSLs) for residential soil (USEPA, 2017) and the Arizona SRLs (AZ, 2007). Worksheet #15 of this UFP-QAPP provides a list of the PALs for each constituent that will be analyzed.

### 3. Who will use the data and what will the data be used for?

USCBP, USACE, ADEQ, USEPA, and the Project Team will use the data for the following:

Following excavation, confirmation soil samples will be collected to compare the results with the PALs. Additional information regarding confirmation soil sampling and decision points are included in Worksheet #14, under Confirmation Sampling. Stockpile soil sampling will be conducted to characterize the waste and select the appropriate disposal facility.

#### **4. What quality of data is required to support an environmental decision?**

All analytical methods completed in the laboratory are planned to be definitive quality data as described in Worksheet #20. Definitive data are defined as data that are suitable for final decision making. They are generated using rigorous analytical methods. Definitive data are not restricted in their use unless quality problems require data qualification, resulting in unusable data. All data will be verified/validated in accordance with the corresponding worksheets.

Data will need to meet the requirements defined in this UFP-QAPP to meet project objectives. Refer to Worksheet #15 for discussion of the target compound reporting levels as they relate to the project-specific action level objectives. Refer to Worksheet #36 for complete details regarding data verification.

#### **5. Are there any special data quality needs, field or laboratory, in order to support the environmental decisions?**

There are no special data quality needs.

#### **6. Where, when, and how should the data be collected/generated?**

Field mobilization will begin following completion of all project plans, the public notice, and the public meeting. Samples will be collected according to SOPs defined in Worksheet #21 and provided in Appendix A. Samples will be handled and shipped according to SOPs #2 through #6 (also referenced in Worksheet #21). Analytical SOPs are referenced in Worksheet #23.

#### **7. Who will collect and generate the data?**

The Project Team will collect the soil samples and generate field data. Analytical data will be generated by an offsite subcontracted laboratory, Test America – Denver.

#### **8. How will the data be reported?**

The data will be evaluated and reported in the RACR.

#### **9. How will the data be archived?**

All field data will be collected on the appropriate field forms or logbook and placed with the project files. All analytical data will be stored on the Sol-JCP servers. Raw data, as well as data summary tables, will be included in the RACR. Hardcopy data will be released to the USACE following completion of the project.

## Worksheet #12-1 – Measurement of Performance Criteria Table

**Matrix:** Soil

**Analytical Group:** PAHs

**Analytical Method:** 8270D

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)
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	PAH	One MS/MSD per 20 samples	Accuracy/Bias/Precision	Quality Systems Manual (QSM) or Laboratory % Recovery / Relative Percent Difference (RPD) Control Limits.	S & A
Method Blank	PAH	One per preparatory batch (20 samples)	Accuracy/Bias-Contamination	No Target Compounds > 1/2 LOQ; no common lab contaminants > LOQ.	A
Laboratory Control Sample (LCS)	PAH	One per preparatory batch (20 samples)	Accuracy/Bias	QSM or Laboratory % Recovery Control Limits	A
Internal Standards	PAH	Each calibration standard, sample and QC sample	NA	EPA Method Requirements	A
Surrogates	PAH	Every field and QC sample	Accuracy/Bias	QSM or Laboratory % Recovery Control Limits	A
Field Duplicate	PAH	One field duplicate per 10 field samples	Precision	For values greater than the LOQ, %RPD ≤ 50%	S & A

## Worksheet #12-2 – Measurement of Performance Criteria Table

**Matrix:** Soil

**Analytical Group:** Metals

**Analytical Method:** 6010C

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)
MS/MSD	Metals	One MS/MSD per 20 samples	Accuracy/Bias/Precision	QSM or Laboratory % Recovery / RPD Control Limits.	S & A
Method Blank	Metals	One per preparatory batch (20 samples)	Accuracy/Bias-Contamination	No Target Compounds > 1/2 LOQ; no common lab contaminants > LOQ.	A
LCS	Metals	One per preparatory batch (20 samples)	Accuracy/Bias	QSM or Laboratory % Recovery Control Limits	A
Dilution test	Metals	One per preparatory batch if MS or MSD fails. Only applicable for samples with concentrations > 50 x LOQ.	Accuracy/Bias/Precision	N/A	A
Post digestion spike addition	Metals	When dilution test fails or analyte concentration of all samples < 50 x LOQ	Accuracy/Bias	N/A	A
Field Duplicate	Metals	One field duplicate per 10 field samples	Precision	For values greater than the LOQ, %RPD ≤ 50%	S & A

## Worksheet #13 – Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (data types, data generation/ collection dates)	How Data Will Be Used	Limitations on Data Use
Soil	TerranearPMC, <i>Final Remedial Investigation / Feasibility Study</i> , 2014.	soil analytical results	Historical data used to determine approximate excavation depths and area.	None
Soil	HDR, Background Arsenic Investigation Report, 2018.	Soil analytical results.	Used to determine background arsenic levels at the site.	None.



## Worksheet #14 – Summary of Project Tasks

This UFP-QAPP focuses on the following field and project-related activities:

- Pre-Construction Activities
  - Site Security
  - Mobilization and Site Preparation
  - Truck Ingress/Egress
  - Survey
  - Utility Location
  - Clearing of Work Area
  - Removal of Structures
  - Erosion and Sediment Control
- Soil Excavation and Temporary Stockpiling
  - Soil Excavation
  - Temporary Stockpiles
  - Confirmatory Soil Sampling
- Disposal and Site Restoration
  - Waste Characterization
  - Disposal
  - Decontamination Procedures
  - Site Restoration
  - Demobilization
- Project-related activities
  - Laboratory analytical tasks
  - Data management and storage
  - Data review
  - Reporting

### Pre Construction Activities

The following activities will be completed prior to any intrusive work.

#### Site Security

Security of former USCBP Firing Range will be maintained through-out the duration of the remediation. Remediation personnel shall coordinate with the USCBP to ensure compliance with all security measures. The only persons with permitted access to the former USCBP Firing Range are the USCBP staff and the property owners. A potential does exist for trespassers to enter the area. Additionally, fire-fighting personnel and equipment may be required to enter the site to suppress brush fires.

Because there is no fencing or other restrictions to the area, the following zones will be established at the Site. The Exclusion Zone (EZ), Contamination Reduction Zone (CRZ), and Support Zone (SZ) as illustrated in Figure 4 will be established as described below:

- Exclusion Zone (EZ): includes areas where potentially contaminated materials will be excavated or handled and all areas where contaminated equipment or personnel may travel. The EZ also includes an equipment laydown area when not in use. Orange fencing will be installed around the perimeter of the EZ as well as a Project Sign and warning placards. This will provide a physical separation that serves as the transition between the EZ and SZ for the transfer of construction materials and equipment from off-site to the EZ, decontamination of vehicles prior to reentering the SZ, decontamination of personnel and clothing (including containerization of disposable outerwear), and personnel decontamination facilities.
- Contamination Reduction Zone (CRZ): A personal hygiene and decontamination station will be set up in the CRZ for personnel to remove contaminated personal protective equipment (PPE) and to wash when exiting the EZ. An equipment decontamination station will be set up in the CRZ for equipment to be decontaminated when exiting the EZ.
- Support Zone (SZ): provides a location for temporary site facilities and an entry and exit area for personnel, material, and equipment from the project site.

### **Mobilization and Site Preparation**

Prior to mobilizing equipment, the work area will be inspected by Sol-JCP and the USACE representative to identify existing conditions. Areas of concern, such as trees, shrubs or structures that need to be protected, will be identified and marked. A series of digital color photographs will be taken to establish the pre-field activity site conditions. Sol-JCP will include the results of this survey in the subsequent monthly progress report / invoice. Sol-JCP will avoid those environmental features included in the survey report and any indicated on the drawings. A series of digital color photographs will be taken to establish the pre-field activity site conditions. Additional photographs will be taken during the project and submitted to the USACE as part of the monthly progress report / invoice.

A project staging area will be established with portable toilets and hand wash stations. Areas for personnel and truck access and egress will also be established with signage for authorized personnel only. Areas specified for decontamination of equipment, the loading and unloading of materials, clean soil storage, and soil stockpiling will be defined prior to commencement of work. The area of excavation will be accessed via W. Target Range Road.

As part of community involvement activities required by A.R.S. § 49-176, signage will be displayed on-site during remedial activities. The sign will describe the type of work being done and include contact information for USACE, USCBP, ADEQ, and Sol-JCP. Refer to Appendix C for an example of the site signage.

### **Truck Ingress/Egress**

Paved and dirt surface roads provide access to the Site. Where possible, all vehicle traffic will remain on these roads. A Stabilized Construction Entrance (SCE) may be required during execution of construction activities. The necessity of the SCE will be determined in the field by the Field Team Leader. If required, the SCE will be constructed in accordance with the details provided on Drawing 1. Depending on the condition of access roads prior to and during construction, improvements and/or reconstruction to adequately accommodate the anticipated construction equipment and traffic during construction

activities may be required. In the event that the roads used for site ingress/egress require improvement to accommodate construction or other equipment utilized during the removal action, temporary gravel roads may be installed. If required, the temporary roads will be constructed in accordance with the details provided in the RAWP.

### **Topographic Survey**

A pre-topographic and post-topographic survey will be performed by a registered land surveyor licensed in the state of Arizona. The survey will consist of establishing three permanent control points, and a sufficient number of temporary control points and off-sets to perform for locating and marking the limits of the specified excavation areas and confirmatory sampling grids. The horizontal extent of the excavation will be determined relative to the Arizona Coordinate System of 1983, Central Zone 0202 (which covers Santa Cruz County, Arizona) to the nearest 1.0 foot. Based on the established control points, the excavation area and a 50 foot grid will be marked with paint and/or flags by SOL-JCP personnel.

### **Utility Location**

Upon delineation of the excavation area, Sol-JCP will contact the following parties to locate and mark-out the utilities in the area:

- Arizona811: 800.STAKE.IT (800-782-5348) will be contacted at least two full working days, but not more than 15 calendar days before intrusive activities. Arizona Blue Stake was established as a one-call notification system by underground facility owners such as water, cable, gas, telephone, and electric to assist excavators in notifying underground facility owners prior to digging.
- Any utilities identified will be contacts and request for identification at the property boundary will be made.
- A private utility locating service will be used to mark any utilities located within the excavation area. At this time, no utilities are expected within the excavation area. However, if one is identified, it will be avoided and a contingency plan will be develop for addressing soils within 3 feet on either side of the utility.

### **Clearing of Work Area**

Sol-JCP will remove debris, trash, slash, logs, snags, branches, and brush from the work and excavation area. This woody material will be disposed of on site, as much as possible out of sight of the roadway. Other debris and trash will be separated from woody material and will be properly disposed of with other construction debris. The stockpile area will also be cleared of any debris or large rocks that may interfere with the geomembrane liner.

### **Removal of Structures**

Range structures and concrete pads shown in Figure 3 will be removed and disposed of at a Class III landfill. As the buildings were erected in 1992 and are not insulated, neither lead-based paint nor asbestos is expected to be present. Since the structure is a single-story that would pose no hazards to neighboring structures or personnel, a common demolition plan will be determined in field and will be submitted to the USACE COR for approval. Prior to disposal, any loose soil will be brushed off the building materials.

Demolition debris from firing position structures will be disposed of at Rio Rico Landfill, located in Rio Rico, Arizona.

### **Erosion and Sediment Control**

Soil erosion and sediment controls as well as storm water management controls may be required during execution of construction activities. Field conditions and on-site Sol-JCP personnel will dictate and identify the control techniques and specific locations for installation of such applications. The controls may include, but are not limited to, silt fence, straw bales, and plastic sheeting. The erosion and sediment control practices and stormwater management controls will remain in place during construction activities until deemed no longer necessary by the field engineer. If required, controls will be inspected periodically and maintained throughout the duration of the remedial action.

### **Soil Excavation and Temporary Stockpiling**

Upon the completion of the pre-construction activities described above, the soil will be excavated and stockpiled. During the RI, soil samples collected from four out of five samples had TCLP lead results above 5 mg/L. Based on these results, and the results of the total lead levels observed in the soil, it is anticipated that a majority of the material within the Western portion of the excavation area will be characteristically hazardous and that there is the potential for excavated soil from the Eastern portion of the Site to be non-hazardous. Therefore, as soil is excavated, it will be segregated and stockpiled to await transportation to the appropriate off-site disposal facility.

### **Soil Excavation**

The areas of lead impacted surface soil exceeding the EPA RSL for lead (400 mg/kg) are depicted in the RI figures. Elevated levels of antimony, arsenic, and PAHs are collocated with lead-impacted soil within the excavation area. The excavation area is approximately 0.5 acres and is rectangular in shape, approximately 250 feet by 200 feet. The excavation will begin in the southwest corner and proceed to the northeast corner. During the RI, the Site was characterized in 50 by 50 foot grids as shown on Figure 3. Within each grid, soil will be excavated as follows:

- The Berm area will be excavated in its entirety. Dimensions are assumed to be approximately 120-foot x 25-foot x and average height of 7-foot tall (approximately 1,166 tons).
- Western Portion: 42 inch maximum depth in grids N-7, N-8, N-9, N-12, N-13, N-14, 78, N-16 and N-17 (approximately 5,000 tons).
- Eastern Portion: 3-foot maximum depth in grids N-10, N-61, N-15, N-60, N-18 and N-59 (approximately 2,916 tons).

The excavation will be performed using a backhoe/front-end loader. Excavation depths will be measured using marked rods. Based on the proposed excavation depths, excavations will not require benching or shoring. The field crew will prepare daily reports that will include load counts and approximate volumes.

The presence of nuisance dust will be monitored throughout construction activities using a dust meter (mini-ram). Per the AAP/SSHP (Sol-JCP, 2018b), if dust readings exceed 1 milligrams per cubic meter ( $\text{mg}/\text{m}^3$ ), engineering controls will be implemented to mitigate dust generation. Soil will be hand dug

around any utilities and any other objects that will not be removed from the excavation area. An X-ray fluorescence (XRF) spectrometer will be employed to field screen excavated areas for metals in real-time. The XRF output is in a concentration (mg/kg) which will be compared directly against the Project Action Level for lead to segregate soil for stockpiling. Confirmation samples will be collected from the excavation boundary sidewalls and from the excavation base.

### **Temporary Stockpiles**

Prior to each bucket of soil being placed in the stockpile area, a grab sample will be collected, with a portion analyzed using XRF and a portion being compiled into the stockpile sample bucket. Using the results of the real-time XRF output, excavated soil will be segregated and temporarily stockpiled prior to load-out. Refer to SOP-22 for field screening procedures using a XRF spectrometer. Based on nature of the Site and the results of soil characterization samples collected and analytical for the RI, the soil is anticipated to be characteristically hazardous. Therefore, the XRF output will be compared to the Project Action Level of 400 mg/kg for segregation into potentially hazardous (>400 mg/kg) and potentially non-hazardous stockpiles (<400 mg/kg). Final determination for waste characterization will rely on laboratory analysis.

Stockpiled soil will be placed over polyethylene sheeting with a minimum thickness of 20 millimeters (mils) or other impervious material. Stockpiles will be constructed to prevent incursion of rain or stormwater through the use of wattles. At the end of each workday or when wind speeds exceed 15 miles per hour, stockpiles will be covered. Covers will be weighted down and/or secured with ropes or other devices as necessary to prevent wind or storm damage.

Stockpile maintenance will be performed as necessary throughout the project duration. Use of additional grading, berming, or curbing to prevent runoff of contaminated flows and divert run-on away from these areas will be utilized as a contingency measure if a rain event occurs that demonstrates the need for additional diversion to provide a non-erosive flow velocity.

### **Confirmatory Soil Sampling**

After excavation of the individual designated excavation areas is complete or nearly complete, a 50 foot grid pattern will be established over the entire remediation site and tailored to excavation area. The grid will be constructed using stakes and string lines based on surveyor offsets. The grids will be numerically identified consistent with the RI. The grid will be maintained during confirmation sampling and potential additional remediation or sampling.

Confirmation soil samples will be collected from the outside boundary side walls and excavation floor when the planned extent of excavation has been reached to determine whether or not the horizontal and vertical extent of each excavation is sufficient to meet the Project Action Levels. The confirmatory sampling program will include collecting one discrete (grab) sample per 25 feet of outside boundary side wall beginning at the southwest corner of the excavation and one composite sample per 50 foot grid base, resulting in the collection of 32 sidewall samples and 15 base samples over the 0.5 acre excavation area for XRF screening. The applicable SOP for discrete (grab) soil sampling is provided in SOP-07. The applicable SOP for composite soil sampling is provided in SOP-08. The applicable SOP for field screening procedures using a XRF spectrometer is provided in SOP-22. In addition, 50 percent of the sidewall

samples (16 samples) and 100 percent of the excavation base samples (15 samples) will be submitted to the laboratory and analyzed for antimony, arsenic, lead and PAHs (total of 31 confirmation samples for laboratory analysis).

Results of the laboratory analysis of confirmation samples for lead, antimony, arsenic, and PAHs will be compared to Project Action Levels as identified in Worksheet #15. Based on the results, the following decision rules applied:

- If COC concentrations area are at or below the PALs, then backfill and demobilization may occur.
- If COC concentrations are greater than the PALs, then consultation with the USACE Contracting Officer (CO) is required to authorize Notice to Proceed (NTP) for one of the following options within 14 days of receipt of the final confirmation sample to avoid additional equipment rental and other similar costs:
  - Additional excavation until PALs are achieved;
  - Backfilling or leaving site as is and demobilizing until such time additional funding can be obtained to complete the action; or
  - Other to be defined by USACE CO.

## **Disposal and Site Restoration Activities**

Temporarily stockpiled soil will be characterized and scheduled for the load-out and transportation of the soil to the approved facility utilizing licensed haulers as described in this section. Ingress and egress routes will be established so that the trucks can approach the stockpiles without traveling on the impacted soils. Temporarily stockpiled soil will be loaded onto tri-axle trucks using a track-mounted excavator. Construction entrances and proper traffic control will be maintained. Trucks will be inspected to ensure that no soils are transported on the truck tires onto public roadways.

### **Waste Characterization**

Prior to loading, temporarily stockpiled soils will be characterization for disposal at the rate of approximately one sample per 500 cubic yards. This equates to approximately two waste characterization samples from the berm and one waste characterization sample from each grid. During the excavation and stockpiling, one scoop from each excavator bucket will be collected and placed into a 5-gallon bucket and homogenized. A composite sample will be drawn from the representative bucket and analyzed for TCLP lead for pre-disposal waste characterization. Note, waste characterization samples will be submitted with a turn-around-time of 3 days. In addition, prior to disposal, any decontamination water collected will be characterized and disposed of accordingly.

### **Disposal**

All off-site disposal of materials will be performed in accordance with applicable Federal, state, and local regulations pursuant to 40 CFR Part 262, Standards Applicable to Generators of Hazardous Waste ([ 45 FR 33142, May 19, 1980, as amended at 70 FR 10818, Mar. 4, 2005; 81 FR 85724, Nov. 28, 2016] ) and 40 CFR

Part 266, Standards for the Management of Specific Hazardous Wastes and Specific Types of Hazardous Waste Management Facilities (50 FR 666, Jan. 4, 1985).

All soil will be shipped using the current USEPA revised Hazardous Waste Manifest Forms with a copy of each manifest submitted to ADEQ:

- Each shipment will be documented utilizing the Uniform Hazardous Waste Manifest (USEPA Form 8700-22).
- Manifests will be prepared for approval by USACE/USCBP prior to transportation.
- Approved Waste Manifests will require signature by USACE or USCBP authorized personnel prior or the day of transport. Copies of manifests, bills of lading, and weight tickets will be provided to the USACE with the project close-out documents.
- Placards will be affixed to each side and each end of the transport trucks as required by the Department of Transportation.
- Soil that contains TCLP lead at concentrations greater than 5.0 mg/L will require management as a RCRA hazardous waste carrying waste code D008 and shipped to US Ecology located in Beatty, Nevada, which is a RCRA permitted disposal facility. US Ecology treats the waste using a pozzolonic stabilization process that decharacterizes the waste so that it meets the Land Disposal Restrictions (LDR) and can be disposed of in a non-hazardous subtitle D landfill.
- If the TCLP lead concentration in the soil is less than 5.0 mg/L, the soil will be managed as non-hazardous and shipped to the Marana Regional Landfill located in Marana, AZ.

All waste classification forms, waste manifests, and Bills of Lading will be properly completed, signed and maintained. Investigation derived waste (IDW) including PPE, plastic sheeting, decontamination water, and miscellaneous debris will be properly disposed of as solid waste at an approved landfill.

### **Decontamination Procedure**

A pre-fabricated decontamination pad (Spilltech, Model: PAC1225, or equivalent as depicted on Drawing 1) will be installed and utilized as necessary for decontamination of trucks and equipment to remove any excess materials prior to Site departure from the EZ. Based on the size of the excavation, it is anticipated that earth moving equipment will have to enter the area of excavation; thus; tracks, wheels and undercarriage of equipment will require decontamination. All trucks and heavy equipment leaving the sites will be decontaminated prior to departure to prevent release of lead-contaminated materials to the environment. Decontamination will be accomplished by first manual wiping or brushing of surfaces followed by water rinse with tap water stored on site for decontamination purposes. Decontamination water will be containerized and characterized for off-site disposal. At the conclusion of all operations, all equipment will be decontaminated and the decontamination pad will be dismantled by removing the plastic liner and placing it along with fluids and collected soil in the next load of waste to be hauled for disposal.

## **Site Restoration**

Site restoration will follow remediation activities and is limited to backfilling excavated areas with clean soil to restore areas to the prior grade less the berm. In order to avoid the potential for cross-contamination, clean fill will be brought in after contained soils are removed from the Site and the Temporary Stockpile area is dismantled. Clean fill will be stored near the excavation area for placement. Due to the arid climate, revegetation will not be performed as part of site restoration. Imported clean fill will be placed and compacted in lifts no greater than 8-inches (refer to Appendix D for information on the backfill material from CalPortland Plant 101). Water will be used for dust control and compaction to meet 90 percent of laboratory maximum density per ASTM D1557. The excavation limits will be backfilled less the volume of the firing berm resulting in equal or less than in volume and pollutant load from disturbed areas. Upon completion of Site Restoration, a post-topographic survey will be conducted by a licensed surveyor.

## **Demobilization**

At the completion of on-site activities Sol-JCP will conduct demobilization. All equipment and excess materials will be transported off-site. Any remaining trash or debris will be removed. Fencing and signage will be removed. All work area will be inspected and any signs of construction will be removed.

## **Project-Related Activities**

### **Laboratory Analytical Tasks**

Laboratory analytical methods are based on the requirements of this UFP-QAPP and follow laboratory specific, method-compliant SOPs that are referenced in Worksheet #23. The laboratory will analyze all samples for various groups of parameters listed in Worksheet #15.

### **Data Management and Storage**

Both hard copy and electronic data deliverables will be managed for the project. Data collected from field observations and laboratory activities will be stored Sol-JCP's network server. Hard copies of field logs, field forms, chain-of-custody forms, correspondence, and project reports will be maintained in hard copy and electronic format at Sol-JCP's Scottsdale, AZ office. Project data will be retained for five years past the completion of the project. Worksheet #29 provides a brief listing of other project documentation and records.

### **Data Review**

The data verification of the project analytical data will be an ongoing process that is performed by both the analytical laboratory generating the data and Sol-JCP. The initial step of the data verification process will be performed by the analytical laboratory. During this review, the calculations, QC sample data, spike recovery, instrument performance indicators, and project specification will be thoroughly inspected through peer level review prior to its release to the laboratory PM. Any problems or nonconformance issues encountered during the analysis will be noted in the project case narrative that precedes each data

package. Where unexplainable variations appear, calculations will again be checked for errors and the sample collection and analytical procedures reviewed to identify any causes for the inconsistencies. All calculation errors will be corrected and anomalies in the sampling or analytical procedures documented and reported in the project analytical data package. The raw data are then QC reviewed for technical correctness by the laboratory PM before final printing. After the data package has been completed, the transcription of 100 percent of the data is verified by the laboratory Quality Assurance/Quality Control (QA/QC) Manager. The laboratory QA/QC Manager will also review the data for conformance to the project data quality objectives. Sol-JCP will be notified of any existing problems and will be updated as conditions dictate.

The laboratory system for ensuring valid data includes several levels of review. Each level commands specific action to prevent the unqualified release of erroneous data and to correct any problems discovered during the review process. All analytical data generated at the laboratory are extensively checked for accuracy and completeness. The data review process consists of data generation, reduction, and three levels of review, as described below.

All data collected during the project will be reviewed and flagged with the appropriate data qualifiers before reported. Detection limits will vary with sample type and the level of interferences associated with the sample matrix. If anomalous results are obtained, every effort will be made to identify any problems in the sample collection, sample preparation, and/or analysis that could have contributed to the anomaly. If any problems have occurred, they will be reported and will include the results, and the appropriate qualifier, with an estimate of the impact the problem may have had on the data. If the sample results do not conform to the data quality objectives, the data will be thoroughly reviewed in order to identify any existing problems and the sample analysis will be repeated if deemed necessary.

Following the analytical laboratory data review, the sample data will be submitted to Sol-JCP who will be responsible for the review and to compare all data with the project data requirements. Data validation will be completed by a third-party data validation subcontractor. Validation will consist of predominantly EPA Stage 4 data review. Refer to Worksheet #36 for additional information.

## **Reporting**

After the completion of the remediation activities, Sol-JCP will prepare a draft and final RACR. This report will include a summary of project activities, maps showing excavation and sampling areas, sample results, survey date, a discussion of the final disposition of materials, and copies of manifests, bills of lading, weight tickets, photographs, and as-built drawings. The draft report will be submitted to the COR within 60 calendar days of completion of field activities. Within 30 calendar days after receipt of all comments on the draft final report, the Contractor shall revise the report and submit the Final RACR.

## Worksheet #15 – Reference Limits and Evaluation Table

One of the primary goals of this UFP-QAPP is to select the appropriate analytical methods to achieve the Limit of Quantitation (LOQ), Limit of Detection (LOD), and Detection Limit (DL) in order to satisfy the overall product quality objectives (PQOs) (as defined in Worksheet #11 of this UFP-QAPP).

The LOQ is the smallest concentration that produces a quantitative result with known and recorded precision and bias. For DOD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.

The LOD is the smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.

The DL is the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.

The LOQ should be below the PAL to support project decision-making. To determine whether the LOQ will meet the PALs, the DL, LOD, and LOQ have been compared with the PALs, which were derived from the EPA RSLs, the ADEQ SRLs, and the ADEQ Groundwater Protection Levels (GPLs). For arsenic, the PAL is based on the ADEQ SRL of 10 mg/kg in accordance with Arizona Administrative Code (A.A.C.) R18-7-201 et seq. This is also within the background range of 5.7-11 mg/kg identified in the Background Arsenic Investigation Report (HDR, 2018).

The following tables indicate that the LOQ for each listed target will meet the PAL objectives for the soil analytes.

**Matrix: Soil**

Parameter	CAS Number	Units	PALs			QL Goal	Laboratory Limits		
			USEPA RSLs <sup>a</sup>	ADEQ SRLs <sup>b</sup>	ADEQ GPLs <sup>c</sup>		LOQ - Limit	LOD - Limit	DL - Limit
PAHs by 8270									
1-Methylnaphthalene	90-12-0	ug/kg	18,000	--		1,000	5.00	0.260	0.500
2-Methylnaphthalene	91-57-6	ug/kg	240,000	--		1,000	5.00	0.309	0.667
Acenaphthene	83-32-9	ug/kg	3,600,000	3,700,000		1,000	5.00	0.160	0.267
Acenaphthylene	208-96-8	ug/kg	(d)	(d)		1,000	5.00	0.170	0.667
Anthracene	120-12-7	ug/kg	18,000,000	22,000,000		1,000	5.00	0.720	2.50
Benzo[a]anthracene	56-55-3	ug/kg	1,100	690		100	5.00	0.900	2.50
Benzo[a]pyrene	50-32-8	ug/kg	110	69		10	5.00	0.740	2.50
Benzo[b]fluoranthene	205-99-2	ug/kg	1,100	690		100	5.00	1.20	2.50
Benzo[g,h,i]perylene	191-24-2	ug/kg	(d)	(d)		1,000	5.00	1.10	2.50
Benzo[k]fluoranthene	207-08-9	ug/kg	11,000	6,900		1,000	5.00	1.00	2.50
Chrysene	218-01-9	ug/kg	110,000	68,000		1,000	5.00	1.00	2.50
Dibenz[a,h]anthracene	53-70-3	ug/kg	110	69		10	5.00	1.30	2.50
Fluoranthene	206-44-0	ug/kg	2,400,000	2,300,000		1,000	5.00	1.00	2.50
Fluorene	86-73-7	ug/kg	2,400,000	2,700,000		1,000	5.00	0.470	0.667
Indeno[1,2,3-cd]pyrene	193-39-5	ug/kg	1,100	690		100	5.00	1.10	2.50
Naphthalene	91-20-3	ug/kg	3,800	56,000		100	5.00	0.326	0.667
Phenanthrene	85-01-8	ug/kg	(d)	(d)		1,000	5.00	1.10	2.50
Pyrene	129-00-0	ug/kg	1,800,000	2,300,000		1,000	5.00	1.10	2.50
Metals by 6010									
Antimony	7440-36-0	mg/kg	31	31	35	10	2.00	0.733	1.50
Arsenic	7440-38-2	mg/kg	0.68	10	290	0.5			
Lead	7439-92-1	mg/kg	400	400	290	10	0.900	0.310	0.800

Notes:

CAS = Chemical Abstracts Service

QL = Quantitation Limit

LOQ = Limit of Quantitation

DL = Detection Limit  $\mu\text{g}/\text{kg}$  = microgram(s) per kilogram

$\text{mg}/\text{kg}$  = milligram (s) per kilogram

NA = No Applicable Screening Level

LOD = Limit of Detection

(a) US EPA RSLs for residential soil (June 2017)

(b) ADEQ SRLs for residential soil – Appendix A (updated 2007)

(c) ADEQ GPLs (1996)

(d) These compounds were not detected in historical sampling events and are not primary COCs.

## **Worksheet #16 – Project Schedule/Timeline Table**

The Project Schedule is outlined in the *Project Management Plan* (Sol-JCP, 2017). After approval of the Final UFP-QAPP, Sol-JCP will prepare a public notice and attend a public meeting. At least 30 days must pass between the public meeting and the start of field work. It is anticipated that field work will take three months. The draft documents will be submitted to the government for review within 60 days of the completion of field work.

## Worksheet #17 – Sampling Design and Rationale

Matrix	Depth to Sample	Analysis	Number of Samples	Rationale	Sampling Strategy
Sidewall soil samples	1.5-2 ft bgs (halfway between surface and base of excavation)	- PAHs by 8270 - Metals (lead, antimony) by 6010C	One soil sample per 25 linear feet plus QA/QC samples	To confirm that the lateral extent of soil above PALs has been removed.	Soil sample will be collected from the excavation sidewalls, from a location halfway between the surface elevation and the base of the excavation.. Soil samples will be sent to the laboratory for analysis, sample results on a standard turnaround time (TAT).
Base soil samples	3-4 ft bgs		One composite soil sample per 50 ft x 50 ft grid plus QA/QC samples	To confirm that the vertical extent of soil above PALs has been removed.	Composite soil samples will be collected from the excavation base. Each composite sample will be collected to include soil from 4 locations within each grid. Soil samples will be sent to the laboratory for analysis, sample results on a standard turnaround time (TAT).
Soil – IDW	N/A	- TCLP Metals – Lead	Approximately one composite sample per 500 CY (equates to 2 samples from the berm, and one per grid)	To properly characterize the waste for disposal purposes.	Composite soil sample will be collected for waste characterization purposes. Two samples will be collected from the berm material, and one sample will be collected from the material from each grid. Soil samples will be sent to the laboratory for analysis, sample results on a urgent 3 day turnaround time (TAT).
Aqueous - IDW	N/A	- PAHs - Metals (lead, antimony) by 6010C	One sample.	To properly characterize the waste for disposal purposes.	Aqueous samples will be sent to the laboratory for analysis, sample results on a standard turnaround time (TAT).

Notes: The following methods will be used for analysis:  
 PAHs by 8270D  
 Metals (including antimony, arsenic, and lead) by 6010C

## Worksheet #18 – Sampling Location and Methods

Sample Location	ID Number	Matrix	Depth	Analytical Group	No. Samples <sup>1</sup>	Sampling SOP Reference
Sidewall Samples	SW-Sample#	Soil	1-2 ft bgs	Lead, antimony, arsenic, PAHs	16 soil samples	SOP-7 Soil Sampling
Base Samples	B-Sample#	Soil	3-4 ft bgs	Lead, antimony, arsenic, PAHs	15 soil samples	SOP-7 Soil Sampling SOP- 8 Composite Soil Sampling

Notes:

<sup>1</sup>Sample quantities are subject to change based on field conditions. The samples will be collected at the required frequency, as included in Worksheet #17.

## Worksheet #19 and #30 – Sample Containers, Preservation, and Hold Times

Analyte/ Analyte Group	Matrix	Method/ SOP	Container(s)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround <sup>1</sup>
PAHs	Soil	Method 8270 SIM SOP DV-MS-0002 / DV-OP-0015	4 oz glass jar	Cool ≤ 6°C	14 days to extract	40 days from extract	Standard
Metals (lead, antimony, arsenic)	Soil	Method 6010 SOP DV-MT-0021 / DV-IP-0015	4 oz glass jar	Cool ≤ 6°C	180 days	180 days	Standard
Lead	Soil	Method 6200 SOP 022 (XRF)	XRF Sample Cup	None	180 days	180 days	Not Applicable
TCLP Metals – Lead	Soil – IDW	Method 6010 SOP DV-IP-0012	8oz glass jars	Cool ≤ 6°C	180 days Mercury: 28 Days	180 days Mercury: 28 Days	3 day TAT
PAHs	Aqueous – IDW	Method 8270 SIM SOP DV-MS-0002 / DV-OP-0006, DV-OP- 0007	2 x 1 liter, amber	Cool ≤ 6°C	7 Days to extract	40 days from extract	Standard
Metals (lead, antimony, arsenic)		Method 6010 SOP DV-MT-0021 / DV-IP-0010	250mL, HDPE	HNO <sub>3</sub> , pH < 2	180 days	180 days	Standard

Notes:

<sup>1</sup>Standard data package turnaround time is 10 days. Expedited analysis (3 to 5-day TAT) will be requested for soil confirmation samples, as needed.

## Worksheet #20 – Field Quality Control Sample Summary Table

<b>Matrix</b>	<b>Analytical Group</b>	<b>Event</b>	<b>No. of Samples to be Collected<sup>1</sup></b>	<b>No. of Field Duplicates</b>	<b>No. of MS/MSDs</b>	<b>Total No. of Samples to Lab</b>
Soil	Lead, antimony, arsenic, PAHs	Soil	31	4	2	37

Notes:

<sup>1</sup>Sample quantities are subject to change based on field conditions (see Worksheet #14, 17, and 18 of this UFP-QAPP).  
 Field duplicates will be collected at a frequency of one per 10 samples.  
 MS/MSDs will be collected at a frequency of one per 20 samples.

## Worksheet #21 – Project Sampling SOP Reference Table

The field SOPs associated with the project sampling (including, but not limited to, sample collection and sample handling and custody) are listed in the following table. The actual field SOPs are provided in Appendix A.

SOP #	Title	Originating Organization	Equipment Type	Modified for Project?	Comments
SOP-01	Chain of Custody	Sol-JCP	NA	No	This SOP establishes the method and responsibilities associated with the maintenance and custody of samples.
SOP-02	Field Activity Documentation	Sol-JCP	NA	No	This SOP defines the minimum requirements for documenting field activities in the field logbooks.
SOP-03	Sample Handling, Packaging, and Shipping	Sol-JCP	NA	No	This SOP outlines the methods and responsibilities for field personnel to use in the packaging and shipping of environmental samples for chemical and physical analysis.
SOP-04	Sample Labeling	Sol-JCP	NA	No	This SOP establishes guidelines and procedures for sample labeling.
SOP-05	Sample Numbering	Sol-JCP	NA	No	This SOP establishes guidelines and procedures for sample numbering.
SOP-06	On-site Sample Storage	Sol-JCP	NA	No	This SOP establishes guidelines and procedures for on-site sample storage.
SOP-07	Soil Sampling	Sol-JCP	Varies	Yes	This SOP establishes guidelines and procedures for soil sampling.
SOP-08	Composite Sample Preparation	Sol-JCP	Varies	Yes	This SOP describes the requirements for compositing techniques.
SOP-09	Calibration and Maintenance of Measuring and Test Equipment.	Sol-JCP	Varies	No	This SOP establishes the methods and responsibilities associated with the calibration, control, and maintenance of measuring and test equipment.
SOP-10	Field Instrument QA/QC	Sol-JCP	Varies	No	This SOP defines field requirements for QA/QC, for equipment and instrument calibration, inspection, and maintenance.
SOP-12	Field Equipment Decontamination	Sol-JCP	Varies	Yes	This SOP describes the procedures required for decontamination of field equipment.

<b>SOP #</b>	<b>Title</b>	<b>Originating Organization</b>	<b>Equipment Type</b>	<b>Modified for Project?</b>	<b>Comments</b>
SOP-13	Drilling, Development, and Heavy Equipment Decontamination	Sol-JCP	Varies	No	This SOP establishes guidelines for use by field personnel in the decontamination of drilling, development, and heavy equipment.
SOP-18	Lithologic Logging	Sol-JCP	Varies	No	This SOP establishes guidelines and procedures for borehole and sample logging.
SOP-20	Field QC Sampling	Sol-JCP	Varies	No	This SOP establishes guidelines and procedures for conducting field QC sampling.
SOP-21	Management of IDW	Sol-JCP	NA	No	This SOP determines the proper hazardous waste characterization and ensures the waste is disposed of consistent with all applicable Solid and Hazardous Waste Regulations.
SOP-22	Field Screening Using XRF Technology	Sol-JCP	XRF Spectrometer	No	This SOP provides procedures for soil screening using a XRF spectrometer.

## Worksheet #22 – Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field equipment and instruments to be used during field activities and requiring calibration, maintenance, testing, or inspection are included in the table below. Also reference specific SOPs for applicable information.

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Reference	Responsible Person
XRF	Calibrated by the manufacturer	Return to supplier for repair or replacement if not functionin.	Ensure battery is charged. Check warning lights.	Prior to each use	Daily prior to use	Warning lights are off and unit is functioning properly	If there are errors, turn the meter off and then on again to see if the warming lights appear. If they do, contact the manufacturer regarding the issue	SOP-10 SOP-22	Field Team Leader
PPE	No calibration required.	Store PPE in a dry and enclosed area which is free of chemical supplies and potentially contaminated material. PPE, should be stored away from direct sunlight to reduce degradation of sensitive material. Refer	Testing of PPE will be performed in accordance with the APP/SSHP.	Inspection of PPE will be performed in accordance with the APP/SSHP.	Refer to the APP/SSHP for specific testing and inspection frequencies .	Refer to the APP/SSHP for PPE acceptance criteria.	If the PPE fails testing or inspection, do not use and replace.	None	Field Team Leader

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Reference	Responsible Person
		to the APP/SSHP for detailed PPE maintenance procedures.							
Decontamination supplies	No calibration required.	Store decontamination supplies in a dry and enclosed area which is free of non-decontamination related chemicals and potentially contaminated material.	No testing required.	The equipment will be inspected for defects and general cleanliness.	Prior to each use.	Free of cross contamination and dirt.	Replace defective or dirty decontamination equipment with clean equipment.	SOP-12	Field Team Leader
Sample Bottleware	No calibration required.	Sample bottleware will be stored in a dry and enclosed storage unit free of potentially contaminated material. Bottleware shall remain unopened until samples are collected. Pre-preserved bottles containing acids and bases will be stored separately.	No testing required.	The equipment will be inspected for defects, open containers, foreign objects, and breakage.	Prior to each use.	Free of defects, unopened, no visual dirt or abnormal odors.	Do not use and return defective, opened, or potentially unclean bottleware material to the supplier.	SOP-03	Field Team Leader

<b>Field Equipment</b>	<b>Calibration Activity</b>	<b>Maintenance Activity</b>	<b>Testing Activity</b>	<b>Inspection Activity</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>SOP Reference</b>	<b>Responsible Person</b>
Various Supporting Consumable Equipment (i.e. re-sealable bags and sample coolers)	No calibration required.	Store consumable equipment in a dry and enclosed area which is free of non-decontamination related chemicals and potentially contaminated material.	No testing required.	The equipment will be inspected for defects and general cleanliness.	Upon receipt of material.	Free of defects and unopened.	Return defective or opened material to the supplier.	None	Field Team Leader

## Worksheet #23 – Analytical SOP References Table

Analytical SOPs are included in Appendix B.

Reference	Title	Definitive or Screening Data	Matrix/Analytical Group	Equipment Type	Modified for Project? Y/N
DV-MS-0002	Polynuclear Aromatic Hydrocarbons by Gas Chromatography/Mass Spectrometry (GC/MS) Selected Ion Monitoring (SIM) [SW 846 Method 8270C and 8270D]	Definitive	Soil / PAHs  IDW Aqueous / PAHs	GC/MS	N
DV-MT-0021	Inductively Coupled Plasma (ICP) Analysis for Trace Elements by SW-846 Method 6010C	Definitive	Soil / Metals  IDW Aqueous / Metals	ICP	N
DV-IP-0010	Acid Digestion of Aqueous Samples for Metals Analysis by ICP	Preparation	IDW Aqueous / Metals	N/A	N
DV-IP-0012	Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) [SW846 1311 and 1312]	Preparation	IDW Soil / TCLP Lead	N/A	N
DV-IP-0015	Acid Digestion of Solids (EPA 3050B)	Preparation	Soil / Metals	N/A	N
DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C and EPA 600 Series	Preparation	IDW Aqueous / PAHs	N/A	N
DV-OP-0007	Concentration and Clean-up of Organic Extracts (SW-846 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, and EPA 600 series)	Preparation	IDW Aqueous / PAHs	N/A	N
DV-OP-0015	Microwave Extraction of Solid Samples by Method [SW 3546]	Preparation	Soil / PAHs	N/A	N

## Worksheet #24 – Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
GC/MS (PAHs)	Tune Check - Check of mass assignments using PFTBA autotune	Prior to ICAL and daily	Acceptable mass assignments using auto tune function	Retune instrument and verify.	Analyst / Section Supervisor	DV-MS-0002
GC/MS (PAHs)	Initial Calibration (ICAL) Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit.	Initial calibration prior to sample analysis	Each analyte must meet one of the three options below: - Option 1: Relative Standard Deviation (RSD) for each analyte $\leq 15\%$ - Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$ ; - Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$ .	Verify standard solutions still valid, perform instrument maintenance as needed, then repeat the ICAL.	Analyst / Section Supervisor	DV-MS-0002
GC/MS (PAHs)	Initial Calibration Verification (ICV)	Second source standard, once after each ICAL.	All reported analytes within $\pm 20\%$ of true value. If analyte identified as a poor performer (Naphthalene, Indeno(123-cd)pyrene, Dibenz(a,h)anthracene, Benzo(ghi)perylene, d <sub>5</sub> -Nitrobenzene) use criteria of $\pm 25\%$ of true value.	Correct problem, and verify second source standard. Rerun verification. If still fails, repeat initial calibration.	Analyst / Section Supervisor	DV-MS-0002
GC/MS (PAHs)	Retention Time Window Position Establishment	Once per ICAL, and at the beginning of the analytical sequence for each analyte and surrogate.	Set position using the mid-point standard of the ICAL when ICAL is performed. On days when ICAL is not performed, use initial CCV.	NA	Analyst / Section Supervisor	DV-MS-0002

<b>Instrument</b>	<b>Calibration Procedure</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action (CA)</b>	<b>Title/position responsible for Corrective Action</b>	<b>SOP Reference</b>
GC/MS (PAHs)	Continuing calibration verification (CCV)	Daily, prior to sample analysis and after every 12 hours of analysis time; and at the end of the analytical batch.	<p>All reported analytes and surrogates within <math>\pm 20\%</math> of true value. If analyte identified as a poor performer in Table 15, use criteria of <math>\pm 30\%</math> of true value.</p> <p>All reported analytes (except poor performers identified in Table 15) and surrogates within <math>\pm 50\%</math> for end of analytical batch CCV.</p>	<p>Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment. For closing CCVs, if compounds are not identified as critical compounds of concern report results with qualifiers. For closing CCVs, if the compound is identified as a critical compound of concern, then recalibrate, and reanalyze all affected samples since the last acceptable CCV;</p> <p>or</p> <p>Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.</p>	Analyst / Section Supervisor	DV-MS-0002
GC/MS (PAHs)	Internal Standards	During acquisition of calibration standard.	Retention time within $\pm 30$ seconds from retention time of the midpoint standard in the ICAL; EICP area within -	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples	Analyst / Section Supervisor	DV-MS-0002

<b>Instrument</b>	<b>Calibration Procedure</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action (CA)</b>	<b>Title/position responsible for Corrective Action</b>	<b>SOP Reference</b>
			50% to +100% of ICAL midpoint standard.	analyzed while system was malfunctioning		
ICP (Metals)	Initial Calibration (ICAL) - Minimum of one high standard and a calibration blank	Prior to sample analysis.	NA	NA	Analyst / Section Supervisor	DV-MT-0021
ICP (Metals)	Initial calibration verification (ICV)	Second source standard immediately following ICAL	All reported analytes $\pm$ 10% of expected value.	Correct any problems and rerun ICV. If that fails, correct problem and repeat ICAL. No samples shall be analyzed until the second-source calibration verification is successful.	Analyst / Section Supervisor	DV-MT-0021
ICP (Metals)	Low-Level Calibration Check Standard $\leq$ LOQ (Low-level ICV)	Daily after one-point ICAL	All reported analytes must be within $\pm$ 20% of expected value.	Correct any problems, then reanalyze or repeat ICAL. Results cannot be reported without a valid low-level calibration check standard.	Analyst / Section Supervisor	DV-MT-0021
ICP (Metals)	Interference Check Solutions (ICS)	After ICAL and prior to sample analysis	<u>ICS-A</u> : Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); <u>ICS-AB</u> : Within $\pm$ 20% of true value. (Not needed if instrument can read negative responses.)	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	Analyst / Section Supervisor	DV-MT-0021

<b>Instrument</b>	<b>Calibration Procedure</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action (CA)</b>	<b>Title/position responsible for Corrective Action</b>	<b>SOP Reference</b>
ICP (Metals)	Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the sequence.	All reported analytes $\pm$ 10% of expected value.	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment with written approval from the client. or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst / Section Supervisor	DV-MT-0021
ICP (Metals)	Initial and Continuing Calibration Blank (ICB, CCB)	Before analyzing samples, after every 10 field samples, and at the end of the analysis sequence.	No analytes detected > LOD	Correct any problems and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. CCB failures due to carryover may not require an ICAL.	Analyst / Section Supervisor	DV-MT-0021

## Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

All laboratory analytical equipment will undergo maintenance and testing procedure according to the laboratory SOPs. A general guideline to laboratory instrument and equipment maintenance, testing and inspection is provided below. Field instrumentation and equipment maintenance, testing and inspection are defined in SOPs provided in Worksheet #22.

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
GC-MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	Analyst	Quality Assurance Manual – Section 20
GC-MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Response factors and chromatogram review	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst	Quality Assurance Manual – Section 20
ICP	Replace pump windings and gas tanks, check standard and sample flow	Monitor ISTD counts for variation	Instrument performance and sensitivity	As needed	Monitor ISTD counts for variation	Replace windings, recalibrate and reanalyze	Analyst	Quality Assurance Manual – Section 20
Microwave Extraction	Power measurement calibration	Performance check	Instrument performance and power	Annually or as needed	Pass manufacturer's criteria	Field service	Vendor (by Contract)	SOP DV-QA-0015 – Section 10.21

## **Worksheet #26 – Sample Handling System**

To verify sample authenticity and data defensibility, a proper sample handling system will be followed from the time of sample collection to final sample disposal.

The Field Team Leader or designee will be responsible for the sample collection, sample packing, and coordination of sample shipment. The samples will be sent to the subcontracted ELAP and Arizona certified laboratory (Test America – Denver) via FedEx overnight. Refer to Appendix B, SOP-03 for the Sample Handling, Packaging, and Shipping SOP.

## Worksheet #27 – Sample Custody Requirements

Proper sample handling, shipment, and maintenance of a chain-of-custody are key components of building the documentation and support for data that can be used to make project decisions. It is important that sample handling and sample chain-of-custody requirements be performed completely, accurately, and consistently.

The specific sampling location of each sample will be recorded with each sample identification number in the field log book and on the chain-of-custody record. The chain-of-custody procedures will be conducted in accordance with Appendix A, SOP-01. Custody of samples must be maintained and documented from the time of sample collection to completion of the analyses. Each sample will be considered to be in the sampler's custody, and the sampler will be personally responsible for the care and custody of the samples until they are delivered to the courier service for delivery to the laboratory.

Samples will be transported as soon as possible after sample collection to the laboratory for analysis. Handling, packaging, and shipping of samples will be done in accordance with SOP-03.

## Worksheet #28 – Laboratory QC Samples Table

The following tables are provided for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level.

Matrix: Solid & Aqueous

Analytical Group: PAHs

Analytical Method/SOP: EPA 8270 / SOP # DV-MS-0002

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	DQI	Measurement Performance Criteria
Internal Standards	Each calibration standard, sample and QC sample	Retention time within $\pm 30$ seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DOD QSM requirements. If field samples still outside criteria, qualify data and explain in case narrative.	Analyst / Section Supervisor	NA	EPA method requirements
Method Blank	One per preparatory batch (20 samples)	No Target Compounds $> \frac{1}{2}$ LOQ and $> 1/10$ the amount in any sample or $1/10$ the regulatory limit (whichever is greater). No common lab contaminants $> LOQ$ .	If sufficient sample is available, reprep and reanalyze samples. Qualify data as needed. Narrate if reanalysis cannot be performed.	Analyst / Section Supervisor	Accuracy/Bias - Contamination	No Target Compounds $> 1/2$ LOQ; no common lab contaminants $> LOQ$ .
LCS	One per preparatory batch (20 samples)	QSM limits (if available) or current in-house limits if no QSM limits published.	Reanalyze LCS once. If acceptable, report. Otherwise, if exceedance is not a critical chemical of concern as identified by the project team, evaluate for sporadic marginal exceedance (SME). If acceptable, report with case narrative comment. If not	Analyst / Section Supervisor	Accuracy/Bias	QSM or Laboratory % Recovery Control Limits

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	DQI	Measurement Performance Criteria
			acceptable for SME, evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.			
Matrix Spike/Matrix Spike Duplicate	One MS/MSD per preparatory batch (20 samples)	Recovery: QSM limits (if available) or current in-house limits if no QSM limits published. RPD: RPD between MS and MSD $\leq$ 20%	Determine root cause; J flag analytes in parent sample if acceptance criteria not met. Discuss in narrative.	Analyst / Section Supervisor	Accuracy/Bias / Precision	QSM or Laboratory % Recovery / RPD Control Limits
Surrogates	Every field and QC sample	QSM limits (if available) or current in-house limits if no QSM limits published.	Evaluate data, if samples non-detect and surrogate recovery is above upper limits, report with case narrative comment. If obvious chromatographic interference is present, report with narrative comment. Otherwise, reextract and reanalyze.	Analyst / Section Supervisor	Accuracy/Bias	QSM or Laboratory % Recovery Control Limits

Matrix: Solid & Aqueous  
 Analytical Group: Metals  
 Analytical Method(s)/SOP: EPA 6010 / SOP # DV-MT-0021

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	DQI	Measurement Performance Criteria
Method Blank	1/ Preparatory Batch (20 samples)	No Target Compounds > ½ LOQ and greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Common lab contaminants: no analytes detected > LOQ.	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >10x blank result or sample results ND.	Analyst / Section Supervisor	Accuracy/Bias - Contamination	No Target Compounds > 1/2 LOQ
LCS	1/Preparatory Batch (20 samples)	QSM limits (if available) or current in-house limits if no QSM limits published.	If acceptable, report. If LCS has high bias, and samples non- detect, report with case narrative comment. If LCS has low bias, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. Marginal exceedance allowed unless analyte is specified risk driver.	Analyst / Section Supervisor	Accuracy/Bias	QSM or Laboratory % Recovery / RPD Control Limits
Matrix Spike/Matrix Spike Duplicate	1 pair/Preparatory Batch (20 samples)	<u>Recovery</u> : QSM limits (if available) or current in-house limits if no QSM limits published. <u>RPD</u> : RPD between MS and MSD ≤ 20%	<b>If MS fails, consult project- specific DQOs and contact client to see if additional measures need to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met.</b>	Analyst / Section Supervisor	Accuracy/Bias / Precision	QSM or Laboratory % Recovery / RPD Control Limits

QC Sample	Number/ Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/position of person responsible for corrective action	DQI	Measurement Performance Criteria
			If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.			
Dilution test	One per preparatory batch if MS or MSD fails. Only applicable for samples with concentrations >50 x LOQ.	Five-fold dilution must agree within $\pm$ 10% of the original determination	If dilution test fails analyze post digestion spike.	Analyst / Section Supervisor	Accuracy/Bias / Precision	N/A
Post digestion spike addition	When dilution test fails or analyte concentration of all samples < 50 x LOQ	Recovery within 80-120% of expected results	For specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Analyst / Section Supervisor	Accuracy/Bias	N/A

## Worksheet #29 – Project Documents and Records

The following table identifies the documents and records that are generated for all aspects of the project including, but not limited to, sample collection and field measurement, onsite and offsite analysis, and data assessment.

<b>Sample Collection Documents and Records</b>	<b>Onsite Analysis Documents and Records</b>	<b>Offsite Analysis Documents and Records</b>	<b>Data Assessment Documents and Records</b>	<b>Others</b>
Field notes Chain-of-custody and shipping records Waste profile information	Field log books Photographic documentation Field change request Equipment maintenance, testing, and inspection logs Daily Equipment Calibration Sampling Data Sheet	Hard copy and electronic analytical data	Data verification and validation reports	APP/SSHP Health and safety tailgate forms Staff health and safety records Activity Hazard Analysis Forms Incident Report Staff & visitor sign in records IDW Tracking Log – Container Delivery and Pickup IDW Tracking Log

Sol-JCP will submit a Quality Control Summary Report (QCSR) documenting the review of analytical chemistry data and the quality and usability of the data collected.

## Worksheet #30 – Analytical Services Table

The following table provides each matrix, analytical group, and concentration level planned for the analytical services. It also identifies all laboratories that will provide analytical services for the project.

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory	Backup Laboratory/ Organization
Soil	PAHs	Low	All samples collected from any location referenced in Worksheet #17.	Reference SOPs defined in Worksheet #23.	5 day TAT	Test America (Denver) 4955 Yarrow Street Arvada, CO 80002	TBD by Sol-JCP if necessary
	Metals (lead, antimony, arsenic)	Low					
IDW Waste (Soils)	RCRA Characteristics	Low	Composite sample from soil stockpiles.	Reference SOPs defined in Worksheet #23.	3 day TAT	Test America (Denver) 4955 Yarrow Street Arvada, CO 80002	TBD by Sol-JCP if necessary
	TCLP Lead	Low					
IDW Waste (Aqueous)	PAHs	Low	Grab sample from containerized liquids (i.e. decon fluids).	Reference SOPs defined in Worksheet #23.	Standard (10 days)	Test America (Denver) 4955 Yarrow Street Arvada, CO 80002	TBD by Sol-JCP if necessary
	Metals (lead, antimony, arsenic)	Low					

## Worksheet #31, #32, and #33 –Assessments and Corrective Action

Assessment Type	Organization Performing Assessment	Frequency	Assessment Deliverable
Data review and verification	Sol-JCP	Per sampling event	RACR
Health and safety review	Sol-JCP QA/QC Officer	As needed	Tailgate Meeting Form
Field Procedures Assessment and UFP-QAPP Compliance	Sol-JCP QA/QC Officer	Weekly	Corrective action report, if needed
Field Sampling Technical Systems Audit	Sol-JCP QA/QC Officer	One at sampling startup	Memorandum
Offsite Laboratory Technical Systems Audit for Test America	American Association of Laboratory Accreditation (A2LA)	Every two years	Letter

## Worksheet #34 – Verification Process Table

Verification is a completeness check that is performed before the data review process continues in order to determine whether the required information (the complete data package) is available for further review. Internal or external is in relation to the data generator.

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain-of-custody and Shipping Forms	Chain-of-custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper’s signature on the chain-of-custody will be initialed by the reviewer, a copy of the chain-of-custody retained in the site file, and the original and remaining copies taped inside the cooler for shipment. See SOP-3 for further details.	Internal	Chris Bason, SOL-JCP QA/QC Officer or Sol-JCP Field Team Leader
Field Log Books	Field notes will be reviewed internally at the end of each working day and placed in the site file.	Internal	Chris Bason, SOL-JCP QA/QC Officer or Sol-JCP Field Team Leader
Laboratory Data	Laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. Received data packages will be verified internally by Sol-JCP.	Internal/External	Test America – Denver  Sol-JCP
Project Reports	Project reporting activities will undergo a QA review by internal Sol-JCP senior staff and/or independent technical reviewers with applicable expertise dependent upon the content of the report.	Internal	Varies, Sol-JCP

## Worksheet #35 – Data Verification Procedures

Verification steps will follow the requirements of this UFP-QAPP and associated SOPs. Data verification and validation is more completely defined in Worksheet #36.

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field Logbook and other miscellaneous field forms	QAPP, Field SOPs	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed and results are documented.	Chris Bason, SOL-JCP QA/QC Officer or Sol-JCP Field Team Leader
Chain of Custody Forms	QAPP, Field SOPs	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Chris Bason, SOL-JCP QA/QC Officer or Sol-JCP Field Team Leader

<b>Records Reviewed</b>	<b>Requirement Documents</b>	<b>Process Description</b>	<b>Responsible Person, Organization</b>
Laboratory Deliverable	QAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the CoCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Program Chemist
Audit Reports, Corrective Action Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan.	Chris Bason, SOL-JCP QA/QC Offier

## Worksheet #36 – Data Validation Procedures

Matrix	Analytical Group	Verification Criteria	Data Validator (Title and Organizational Affiliation)
Soil, sediment, and surface water	All groups	One hundred percent of all sample data (excluding waste characterization data used for disposal facility selection purposes only), unless noted otherwise.	Sol-JCP Team will provide final validated data for all methods.

### Data Verification/Validation Scope Overview

One hundred percent of all samples will be validated. Validation will be completed in accordance with the EPA Guidelines. Data validation will be completed by a third party, and will consist of predominantly EPA Stage 4 data review. Significant errors noted during the data validation review may determine an increase in data validation as part of corrective action based on recommendation of the project chemist and with concurrence of AFCEC. Data validation will include but may not be limited to the following parameters:

- Data completeness
- Holding time
- Instrument calibration, initial and continuing
- LCSs
- Method blanks
- Surrogate spikes
- MS/MSDs
- Laboratory and field duplicates
- Sample results verification

Data flags will be assigned using the QC acceptance limits and procedures defined in the UFP-QAPP. Data flags, and the reason for each flag, will be entered into an electronic database and made available to the data users. Multiple flags are routinely applied to a specific sample method/matrix/analyte combination, but there will only be one final flag. A final flag is applied to the data according to the most conservative of the validation flags.

A data validation report will be provided at the end of the remedial activities to summarize the results of the data validation findings and to present conclusions regarding the usability of the data for project objectives. The report will assess the accuracy, precision, representativeness, comparability, and completeness of the data generated. The report will focus on out of control data results and present a table of noncompliant results that exceeded some QC requirement.

### Field Data Review

Field generated information may include field log books, XRF calibrations and readings, ambient air calibrations and results, sample chain-of-custody forms, shipping documents, sampling observations,

sample labels, and other miscellaneous field observations. All field measurements and or field log information will be entered into field log books and reviewed daily by the field team leader or designee. The designee may be a qualified field geologist, hydrogeologist, engineer, environmental scientist, and/or technician.

### **Laboratory Data Review Requirements**

All analytical data generated by the laboratory will be verified before submittal to Sol-JCP. This internal data review process, which is multi-tiered, will include all aspects of data generation, reduction, and quality control assessment. In each laboratory analytical section, the analyst performing the tests will review 100 percent of the definitive data. After the analyst's review has been completed, 100 percent of the data will be reviewed independently by a senior analyst or by the supervisor of the respective analytical section using the same criteria. The Laboratory QA Manager will perform 100 percent review of the data packages generated for the project.

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

- Sample receipt procedures and conditions
- Sample preparation
- Appropriate SOPs and analytical methodologies
- Accuracy and completeness of analytical results
- Correct interpretation of all raw data, including all manual integrations
- Appropriate application of QC samples and compliance with established control limits
- Verification of data transfers
- Documentation completeness (for example, all anomalies in the preparation and analysis have been identified, appropriate corrective actions have been taken and documented in the case narrative(s), associated data have been appropriately qualified, and anomaly forms are complete)

### **Accuracy and completeness of data deliverables (hard copy and electronic) Laboratory Data Evaluation**

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

## Worksheet #37 – Usability Assessment

This Section describes the procedures/methods/activities that will be used to determine whether data are of the right type, quality, and quantity to support environmental decision making for the project.

The hierarchy of applicable guidance documents for data validation / review is as follows:

- (1) Site-specific Work Plan and/or QAPP
- (2) DOD QSM, version 5.0 (July 2013) - covers any gaps in the WP/QAPP requirements
- (3) EM 200-1-10, June 2005

### Usability Assessment

It is the responsibility of the project chemist and the laboratory to confirm that the data meet the LOQs, LODs, and DLs listed in this UFP-QAPP. During the data validation assessment, nonconformances are documented and data are qualified for use in decision making. The data are determined to be usable by the project chemist based on the requirements of this UFP-QAPP. Data gaps will be present if a sample is not collected, a sample is not analyzed for the requested parameters, or the data are determined to be unusable. The need for further investigation will be determined on a case-by-case basis, depending on whether data can be extrapolated from adjacent sampling locations, and whether or not the results are unnecessary based on the results from adjacent locations. All data are usable as qualified by the data validator, with the exception of rejected data. Estimated and/or biased results are usable. Outliers, if present, can be addressed on a case-by-case basis.

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

The data validation reports will identify precision and accuracy exceedances with respect to the laboratory performance for each batch of samples, as well as comparability of field and lab duplicates. All the results will be assembled and statistically reported for an overall quality assessment provided in the sampling reports and SI report. Discussion will cover precision, accuracy, representativeness, completeness, and comparability (PARCC), defined as follows:

### Precision

If calculated from duplicate measurements:

$$RPD = \frac{(X_1 - X_2) * 100\%}{X_1 + \frac{X_2}{2}}$$

Where:

RPD = relative percent different

X<sub>1</sub> = larger of the two observed values

X<sub>2</sub> = smaller of the two observed values

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

$$RSD = \frac{\text{Standard Deviation}}{\text{Mean of Replicate Analyses}} * 100$$

Where:

$$SD = \sqrt{\frac{\sum_{i=1}^n [(x_i - \bar{x})^2]}{n-1}}$$

SD = standard deviation

$x_i$  = measured value of the  $i$ th replicate

$\bar{x}$  = mean of replicate analyses

$n$  = number of replicates

### Accuracy

For measurements where matrix spikes are used:

$$\%R = \frac{X_1 - X_2}{X_3} * 100\%$$

Where:

%R = spike amount recovered

$X_1$  = spiked sample concentration

$X_2$  = unspiked sample concentration

$X_3$  = spiked concentration added

### Representativeness

Representativeness is a qualitative measure of the degree to which sample data accurately reflects the characteristics of a population of samples. It is achieved through a well-designed sampling program and by using standardized sampling strategies and techniques and analytical procedures. Representativeness is a subjective parameter, and can be achieved by ensuring that the sampling team follows all applicable SOPs for sample collection and handling, and that the laboratory follows all applicable SOPs for sample handling, preparation, and analysis.

### Completeness

Completeness is a measure of the amount of valid data obtained compared with the amount expected under correct, normal conditions. The data completeness objective for this project is 90 percent. It is calculated for the aggregation of data for each analyte measured as a compound of concern for the project objectives. Valid data are data that are usable in the context of the project goals. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set.

### Comparability

Comparability is the extent to which comparisons among different measurements of the same quantity or quality will yield valid conclusions. Comparability among field measurements will be achieved through the use of standard procedures, standard field data sheets, and uniform concentration units. To ensure comparability, field procedures will be standardized and field operations will adhere to SOPs.

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## FIGURES

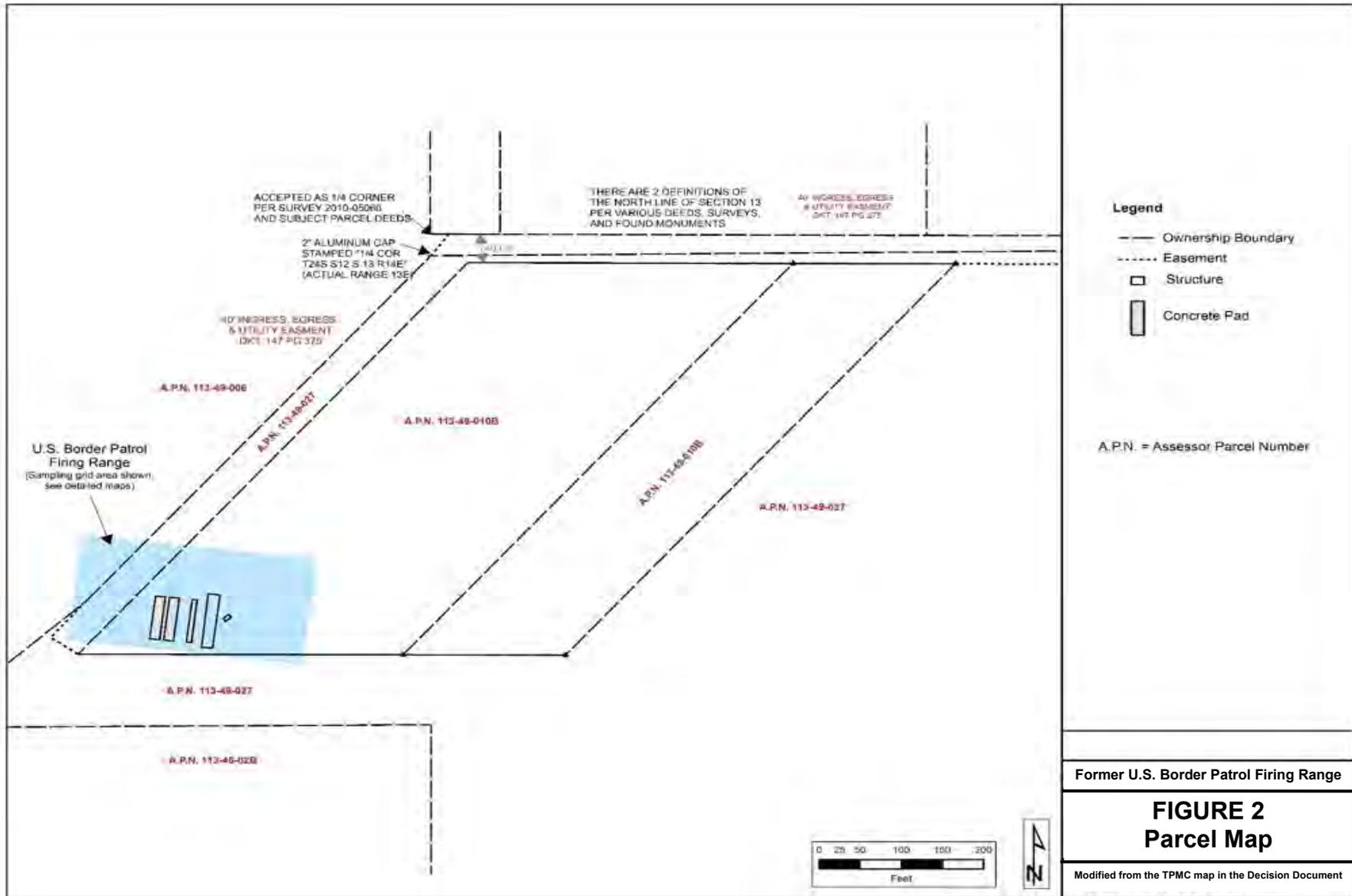


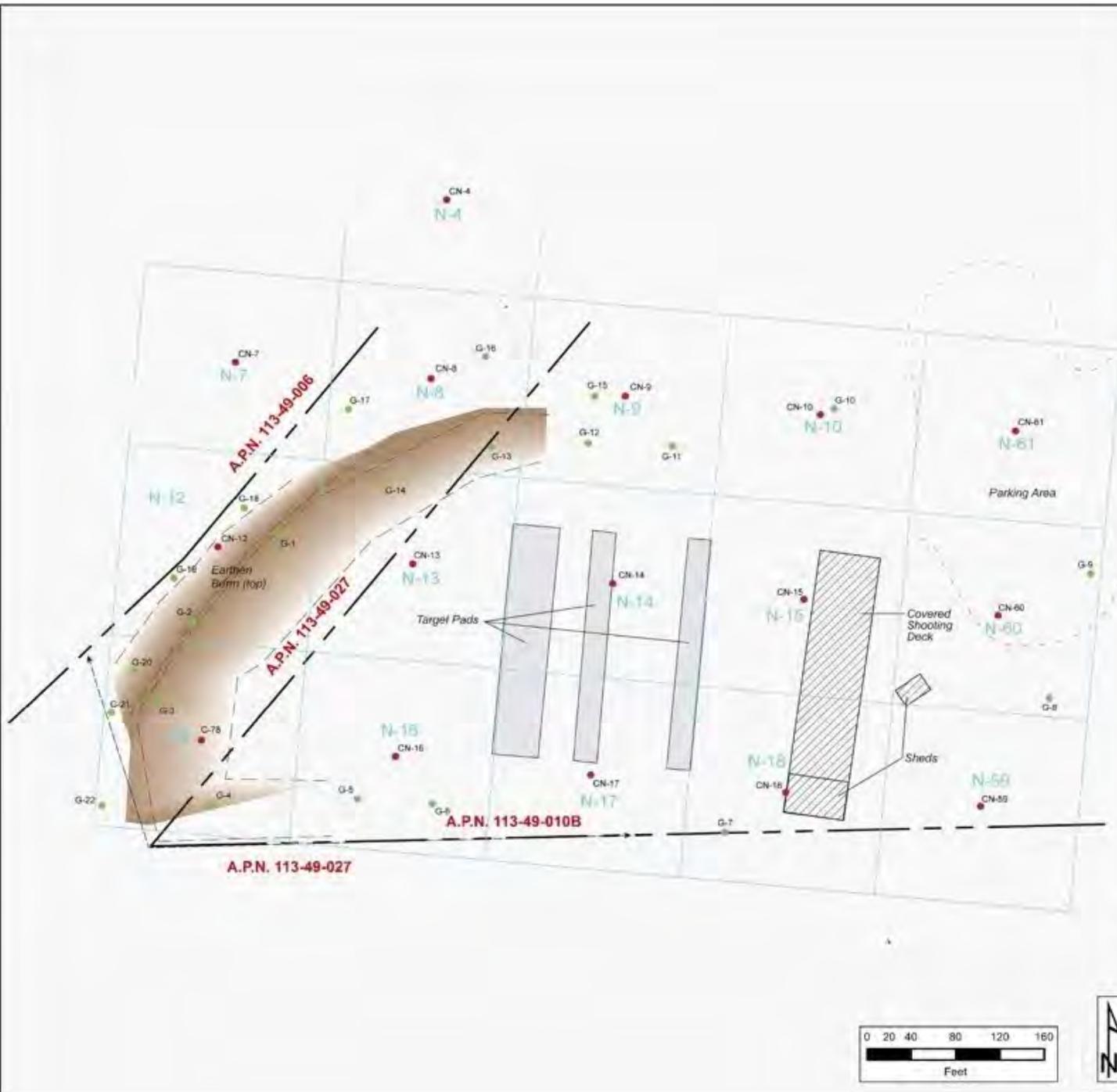
Index Map

Former U.S. Border Patrol Firing Range

# FIGURE 1 Site Location

Date: May 5, 2017  
Modified from TPMC map in the Decision Document





**Legend**

- Grab Sample Location (G-*nn*)
- Composite Sample Location (CX-*NN*)
- Sampling Grid
- - - Ownership Boundary
- Structure (shooting deck, shed)
- Earthen Berm
- Concrete Pad
- - - Fence Line
- - - Parking Lot

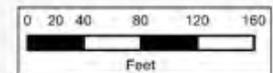
G-10  
G = Grab Sample

CN-10  
N = Grid ID  
C = Composite Sample

Former U.S. Border Patrol Firing Range

**FIGURE 3**  
**2014 RI Sampling**  
**Locations and Grid**

Modified from the TPMC map in the Decision Document





Transportation  
Ingress/Egress  
Route

Support  
Zone

Toilets

Equip

Stockpile  
Area

Contaminant  
Reduction  
Zone

Air  
Monitoring  
Station

Former U.S. Border Patrol Firing Range

**FIGURE 4**  
**Site Layout**

## **APPENDIX A**

### **Field Standard Operating Procedures**

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## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) establishes the method and responsibilities associated with the maintenance and custody of samples which are to be used to provide data which form a basis for making project related decisions. It outlines the general procedures for maintaining and documenting sample chain of custody from the time of sample collection through sample disposition.

## **2.0 REFERENCES**

2.1 USEPA, Test Methods for Evaluating Hazardous Waste, (SW-846) Rev.0, Sept. 1994

## **3.0 RESPONSIBILITIES**

3.1 The Field Team Leader is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Project Chemist is responsible for assuring proper COC is initiated at the time the sample(s) are collected and maintained throughout the handling and subsequent transportation of the sample(s) to the designated laboratory. Additionally, he/she is the project authority for determining the disposition and fate of sample(s) which have identified deficiencies (e.g., missed holding times, elevated temperature at receipt, etc.).

3.3 The Quality Control System Manager (QCSM) is responsible for periodic review of Chain of Custody records are generated documentation associated with this SOP. The QCSM is also responsible for implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to QC sampling requirements, issuing nonconformances, etc.) if problems occur.

3.4 The Field Team Lead(s) is responsible for properly documenting and maintaining the COC from the time of sample collection until the sample is delivered to the lab.

3.5 Laboratory Personnel are responsible for receipt and entry of samples into the laboratory which have been submitted under a COC document. Additionally, samples received will be entered into the laboratory COC procedures by properly documenting and maintaining COC from the moment that they take custody of the sample at the laboratory until the sample is disposed of or returned to the client.

## **4.0 DEFINITIONS/MATERIALS**

### **4.1 Chain of Custody**

The Chain of Custody (COC) document is the written record that traces the sample possession from the time each sample is collected until its final disposition, sometimes called the “cradle to grave” record. Chain of Custody is maintained by compliance with one of the following criteria:

- The sample is in the individual's physical possession
- The sample is maintained in the individual's physical view after being in his/her possession
- The sample is transferred to a designated secure area restricted to authorized personnel
- The sample is sealed and maintained under lock and key to prevent tampering, after having been in physical possession.

#### 4.2 Waybill

A document that contains a list of the goods and shipping instructions relative to a shipment.

#### 4.3 Common Carrier

For the purpose of this procedure, the common carrier is any commercial carrier utilized for the transportation of the sample(s) from the field to the laboratory.

### 5.0 PROCEDURE

#### 5.1 General

5.1.1 An overriding consideration for data resulting from laboratory analyses is the ability to demonstrate that the samples were obtained from the locations stated and that they reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, and laboratory custody until disposal must be documented to accomplish this. Documentation will be accomplished through a COC Record that lists each sample and the individuals performing the sample collection, shipment, and receipt.

5.1.2 The COC document is a preprinted form. The original will accompany the samples lab and a copy will be retained in the field project file.

#### 5.2 Field Sample Custody

5.2.1 Sampling personnel, upon collection of samples for analysis, will properly complete a COC Record form. The COC document will be the controlling document to assure that sample maintenance and custody are maintained thereby assuring the sample(s) are representative of the environment from which they were collected. At a minimum, the following information will be recorded on the COC document:

- The unique identification number assigned to each sample.
- A brief description of the sampling location and a physical description of the sample type.
- The date and time of the sample collection.
- Container type (e.g., glass, poly, brass sleeve, etc.).
- Sample volume and number of containers (e.g., 2 x 40 ml, 3 x 1 liter).
- Sample preservation (e.g., HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, 4°C).
- Requested analyses.
- Special instructions to the laboratory including handling requirements, quality assurance/quality control, health and safety, and sample disposition.
- The project name and number.
- The date the analytical report is due.

- The names of all sampling personnel.
- The name and phone number of the project contact.
- The name and phone number of the laboratory contact.
- The name of the courier and the waybill number (if applicable).
- A unique document reference number.

5.2.2 The COC document will be initiated in the field by the person collecting the sample and signed by each individual who has the samples in their possession. Each time that sample custody is transferred, the former custodian must sign over the COC as Relinquished By, and the new custodian must sign on to the COC as Received By. Each signature must be accompanied by the date, time, and the name of their project or company affiliation.

5.2.3 Transferring of COC from sampling personnel to the analytical laboratory will be performed in accordance with the requirements stated below.

5.2.3.1 If the sampling personnel deliver the samples to the laboratory, transfer of COC occurs as follows:

The sample collector delivers the samples to the laboratory and relinquishes the sample directly to a laboratory representative.

- The collector signs the COC listing his/her name, affiliation, the date, and time. Any person involved in the collection of the sample may act as the sample custodian.
- The laboratory representative must receive the samples by signing his/her name, affiliation, the date, and time on the COC. The laboratory representative may decline to take receipt of the samples if the COC is not properly completed or if the samples are not properly packaged. All designated laboratory personnel may act as the sample custodian.
- One copy of the COC is given to the sample collector to be returned to the project files and one copy is maintained with the samples at the laboratory.

5.2.3.2 If the sampling personnel transfer sample(s) to the laboratory utilizing a common carrier, sampling personnel will retain COC responsibility and the common carrier is not responsible for maintaining sample custody. The sample collectors are responsible for packaging the samples in a manner that meets the COC definition criteria, that is, the samples are sealed to prevent tampering. When transferring samples to the courier for transport, COC procedures are maintained as follows:

- The sample collector lists the courier affiliation and waybill number on the COC.
- The sample collector relinquishes custody by signing his name, affiliation, date, and time. The collector keeps a copy of the relinquished COC for the project file.
- The relinquished original COC is sealed in a watertight plastic bag and taped to the inside of the lid of the container used for transportation.
- The transportation container is sealed to prevent tampering and given to the courier for delivery to the laboratory.
- The sample collector obtains a copy of the waybill from the courier for the project file.

- The laboratory representative must receive the samples by signing his/her name, affiliation, the date, and time on the COC. This copy is maintained with the samples at the laboratory.
- The laboratory representative obtains a copy of the waybill from the courier for the project file.

### 5.3 Analytical Laboratory Custody

5.3.1 Upon receipt at the analytical laboratory, the field generated COC document will be signed, dated, time marked, temperature marked, and laboratory identification will be provided in the appropriate spaces. A sample receipt form will be completed documenting the condition of the samples upon receipt.

5.3.2 Laboratory receipt personnel will enter the samples into the laboratory by implementing the sample custody procedures addressed within their approved Program Plan.

5.3.3 After completion of analytical testing, sample remnants not consumed during testing may be kept for six months beyond the completion of analysis, unless otherwise specified by a notation on the COC that samples are to be returned to the project site for disposal. Once this time period has elapsed, the samples will be disposed of and the disposal record number will be recorded on the laboratory record copy of the COC.

## **6.0 REQUIRED FORMS**

6.1 Chain of Custody Record

6.2 Sample Receipt Form

6.3 Field Log Book

## **1.0 PURPOSE**

The purpose of this Standard Operating Procedure (SOP) is to define the minimum requirements for documenting field activities in the field logbooks. Field logbooks provide a detailed daily handwritten record, kept in real time, of field activities performed at an investigation site. Logbooks are permanently bound by glue or thread into a hard cover, and should be waterproof. Field logbooks may be assigned to specific activities, positions, or areas within the site. Field logbook covers must be sequentially numbered and indicate the position, task, activity, or area assigned to the logbook.

## **2.0 REFERENCES**

None

## **3.0 RESPONSIBILITIES**

3.1 The Field Team Leader is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Field Team Leader is responsible for ensuring that all geotechnical measurements are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.3 The Quality Control System Manager (QCSM) is responsible for periodic review of field generated documentation associated with this SOP. The QCSM is also responsible for implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to QC sampling requirements, issuing nonconformances, etc.) if problems occur.

3.4 The Field Team Lead(s) are responsible for ensuring that field logbooks are completed daily in accordance with this procedure.

3.5 The Sampling Team Members are responsible for making timely and complete entries in the field logbook and for reporting daily activities to the Field Team Leader or QCSM as appropriate.

## **4.0 DEFINITIONS/MATERIALS**

4.1 Field logbook: surveyor's book or field book, bound and ruled/gridded, record book with sequentially numbered and waterproof pages.

4.2 Waterproof black ink pens.

## **5.0 PROCEDURE**

### 5.1 Field Logbook Cover

Label the front cover of the field logbook with the Site name, project number, subcontractor name, Site manager's name and mailing address, client name, the start date and, finish date. The field logbooks must be sequentially numbered.

### 5.2 Field Logbook

The following steps must be followed when making entries in the field logbook:

1. Enter the day and date; time the task started; weather conditions; and the names, titles, and organizations of personnel performing the task.
2. Record the name, title, organization, time of arrival, and time of departure of all visitors to the task area.
3. Describe all site activities in specific detail or indicate which forms were used to record such information (e.g., soil boring log or well completion log). A partial data list is given below:
  - Monitoring wells: complete the Monitoring Well Construction Log form.
  - Monitoring well development: complete the Monitoring Well Development Log
  - Monitoring well purging and sampling: complete the Monitoring Well Purge and Sample Log form.
  - Subsurface soil sampling:
    - Soil borings: complete the Drilling Log and include borehole size depth, sample equipment, method, and samples collected. Detailed lithologic data will be recorded on the boring log.
    - Trenches, and test pits: record the excavation dimensions, sampling equipment or method(s), and samples collected. Detailed lithologic data will be recorded on the Test Pit Information Log.
    - Soil gas and geophysical surveys: grid or line dimensions, probe or sensor spacing, depths, survey and recording equipment type and serial or identification number, and location of resulting data (e.g., strip chart, analog data record, computer file, and file name). Sketches are valuable additions to field notes and should be used where possible.
4. Time of specific field activity, i.e. sample collection, well development, calibration.
5. Describe in specific detail any field tests that were conducted and instruments used. Reference any forms that were used, other data records, and the procedures followed in conducting the test. If the final results of any field activity are obtained in the field, these data should be annotated in the field logbook.
6. Changes in procedures or sample locations and reasons for change.
7. Describe in specific detail any samples collected and whether splits, duplicates, matrix spikes or blanks were prepared.
8. Upgrades or downgrades of personal protective equipment and the rationale for such action, and health and safety information such as level of personal protective equipment (PPE) used.
9. List the time, equipment type, and the procedure followed for all decontaminations carried out. Reference the page number(s) in the decontamination log (if any) where detailed information is recorded; if not referenced, detailed information shall appear in field logbook.

10. List all instrument calibrations, person(s) performing calibration, and the page number of the calibration log that provides specific information on calibration procedures and results when the calibrations occur in the field.
11. Record all photographs by number and include a description of the subject, the direction the photographer is facing, and the photographer's initials. If the event photographed is the collection of a sample, record the sample ID number.
12. List any equipment failures or breakdowns that occurred, together with a brief description of repairs or replacements.
13. No pages may be removed from the site or field logbooks for any reason. Blank pages must be marked "page intentionally left blank".
14. Mistakes must be crossed out with a single line, initialed, and dated. Only persons authorized by the Field Team Leader may make entries in logbooks.
15. The Field Team Leader or Field Team Lead must sign the field logbook at the bottom of each page.

## **6.0 REQUIRED FORMS**

### 6.1 Field Log Book

### 6.2 Any applicable field forms (Soil Sample Collection Field Sheet,)

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) outlines the methods and responsibilities for field personnel to use in the packaging and shipping of environmental samples for chemical and physical analysis. This SOP only applies to the packaging and shipping of limited quantity, low concentration environmental samples. This procedure does not apply to those samples considered hazardous materials, hazardous waste, mixed waste, radioactive waste, and/or dangerous goods. Those requirements are specified in the Department of Transportation (DOT) 49 CFR 114-327 and the International Air Transport Association (IATA) procedures. The details within this SOP are only applicable to the general requirements for sample packaging and shipping and should only be used as a guide for developing more job-specific work plans.

## **2.0 REFERENCES**

2.1 EPA, September 1987, Compendium of Superfund Field Operations Methods , EPA 540/P87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidelines for Conducting Remedial Investigation and Feasibility Studies under CERCLA, Interim Final OSWER Directive 9355.3-01.

2.3 Code of Federal Regulations, DOT 49 CFR parts 100 to 177, Revised October 1, 1992.

2.4 Dangerous Goods Regulations, IATA, January 1, 1994.

## **3.0 RESPONSIBILITIES**

3.1 The Field Team Leader is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Project Chemist is responsible for the development and review of site-specific work plans which address the specific sample handling, packaging, and shipping requirements for the project. Review the project specific documentation forms to ensure they are appropriate for the field activities. The Project Chemist is also responsible for seeing that field personnel receive proper training and maintain quality assurance/quality control (QA/QC). If problems arise, the Project Chemist is responsible for swift implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to requirements, issuing nonconformances).

3.3 The Quality Control System Manager (QCSM) is responsible for the periodic review of documentation generated during sample handling, packaging, and shipping and the periodic review and audit of field personnel as they perform the work.

3.4 The Field Team Lead(s) are responsible for ensuring that samples are handled, packed and shipped in accordance with this procedure

## **4.0 DEFINITIONS/MATERIALS**

#### 4.1 Environmental Sample

A limited quantity, low concentration sample that does not require DOT or IATA hazardous waste labeling as a hazardous waste or material.

#### 4.2 Hazardous Waste Sample

Medium or high concentration sample requiring either DOT or IATA labeling as a hazardous waste or material.

#### 4.3 Hazardous Waste

Any substance listed in 40 CFR Subpart D (260.30 et seq.) or otherwise characterized as ignitable, corrosive, reactive, or toxic as specified in Subpart C (261.20 et seq.) that would be subject to manifest and packaging requirements specified in 40 CFR 262. Hazardous waste is defined and regulated by the U.S. Environmental Protection Agency (USEPA).

#### 4.4 Hazardous Material

A substance or material in a quantity or form which may pose an unreasonable risk to health, safety, and/or property when transported in commerce. Hazardous material is defined and regulated by DOT (49 CFR 173.2 and 172.101) and IATA (Section 4.2).

#### 4.5 Sample

Physical evidence collected from a facility or the environment which is representative of conditions at the point and time at which the sample is collected.

### **5.0 PROCEDURE**

#### 5.1 Sample Handling

5.1.1 Inspect the sampling containers (obtained from the analytical laboratory prior to the sampling event) to ensure that they are appropriate for the samples being collected, correctly preserved, and undamaged.

5.1.2 When collecting a sample always use approved/site specific personal protective equipment (e.g., gloves, etc.) to prevent cross-contamination from sample to sample but also as a health and safety requirement.

#### 5.2 Field Packaging

5.2.1 Collect the samples in accordance with the site-specific work plans and applicable SOPs.

5.2.2 Place all containers in separate, appropriately sized, airtight, seam sealing polyethylene bags (e.g., Ziploc™ or equivalent). Seal the bag, removing any excess air.

5.2.3 Place the bagged container inside an insulating shipping container, “cooler”. This cooler should have ice inside to assure samples remain cool,  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ , during transit from field to the packaging location.

5.2.4 Maintain the samples under chain of custody (COC) in accordance with the site-specific work plans and appropriate SOPs.

### 5.3 Sample Packaging

5.3.1 Inspect the integrity of the shipping container. The container is generally a “cooler” constructed of heavy plastic or metal with appropriate insulating properties so that variations in temperature during shipping are minimized. Also make sure that the drain plug has been sealed with nylon reinforced strapping tape or mailing tape.

5.3.2 Place two to four inches of absorbent packaging material (e.g., Styrofoam bubbles, Vermiculite™ etc.) in the bottom of the shipping container.

5.3.3 Carefully check the COC record against the collected sample labels and containers to ensure that the sample numbers, sample description, date and time of collection, container type and volume, preservative, and the required analytical methods are correct and in agreement.

5.3.4 Place the samples in the shipping container, allowing sufficient room between the samples to place ice and/or packing material.

5.3.5 Double bag (ziplock™ or equivalent) and seal crushed or cubed ice in heavy-duty polyethylene bags. Place these bags of ice on top of and between samples. Include a VOA vial of tap water clearly labeled “temperature blank” so that the laboratory can verify the temperature of the samples upon receipt. The remaining space will be filled with packing material.

5.3.6 All samples requiring temperature preservation stated at 4 °C will be acceptable “as in” within the range of 4°C ± 2°C. The laboratory should record the temperature of receipt upon the COC and complete a cooler receipt form. For all samples received at less than 2°C (note if frozen), or at greater than 6°C, the sample(s) and temperature (in 1°C increments) will be identified on the COC and the Project Chemist notified, to provide a determination and written authorization to proceed to analysis.

### 5.4 Sample Shipping

5.4.1 The laboratory will be contacted 2 weeks prior to sample shipments. Delivery on weekends and holidays will be confirmed in 1 week in advance, and one day prior to shipment. The person in charge of sample custody will time, date, and sign over relinquishment of custody on the COC. When a common carrier is to be used for sample shipment, also record the air/waybill number (tracking number) and the name of the carrier on the COC record. Place the original copy of the COC record in a sealed, clear plastic envelope or bag and tape the COC record envelope to the inside lid of the shipping container. Retain a copy of the COC record for tracking purposes.

5.4.2 Using nylon reinforced strapping tape or mailing tape, seal the shipping container.

5.4.3 Place custody tape over opposite ends of the lid.

5.4.4 Mark the container “THIS END UP”, or apply arrow labels that indicate the proper position to be maintained during shipping. Place a “FRAGILE” label on any cooler containing glass bottles.

5.4.5 Apply a label stating the name and address of the shipper and the receiving laboratory on the outside of the cooler.

5.4.5.1 If QA split samples are shipped. The Project Chemist shall notify the QA Laboratory by telephone at least two weeks in advance of sale shipment (for large numbers of samples, greater than 20 and again on the day that samples are forwarded to the QA Lab.

5.4.6 Turn the sample over to the courier or carrier for delivery to the laboratory. All samples should be shipped by the fastest available method to the laboratory as soon as possible after sample collection.

NOTE: The courier or carrier is not responsible for sample custody and is not required to sign the COC.

5.4.7 Contact the appropriate laboratory personnel to advise them of the sample shipment.

5.4.8 Review the COC and sample collection forms for completeness and turn them over to site or project management.

## **6.0 REQUIRED FORMS**

6.1 Chain of Custody Record

6.2 Sample Receipt Form

6.3 Field Log Book

## SOP-04

**SAMPLE LABELING  
STANDARD OPERATING PROCEDURE****1.0 PURPOSE**

## 1.0 Purpose

This Standard Operating Procedure (SOP) establishes guidelines and procedures for sample labeling. Sample labeling is required to identify, track and trace samples from the time of collection until the time of disposal. Additional specific procedures and requirements will be provided in the project work plans.

**2.0 REFERENCES**

2.1 EPA, September 1987, Compendium of Superfund Field Operations Methods, EPA 540/P87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidance for Conducting Remedial Investigation and Feasibility Studies Under CERCLA, Interim Final OSWER Directive 9355.3-01.

**3.0 RESPONSIBILITIES**

3.1 The Field Team Leader is responsible for ensuring that all sample collection and labeling activities are conducted and documented in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Quality Assurance Control System Manager (QCSM) is responsible for periodic review of field generated documentation associated with this sample labeling SOP. The QCSM is also responsible for the implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to sample labeling requirements, issuing nonconformances, etc.) if problems occur.

3.3 The Field Team Lead(s) assigned to sampling and sample labeling activities are responsible for completing their tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from the procedures to the Field Team Leader.

**4.0 DEFINITIONS/MATERIALS**

## 4.1 Sample Label

Sample labels include all forms of sample identification (labels or tags) that are physically attached to samples collected and provide, at a minimum, the information required by this SOP and project work plans.

## 5.0 PROCEDURE

This section contains the procedures involved with sample labeling. Sample labeling is required to identify, track and trace samples from the time of collection until the time of disposal. The details within this SOP should be used in conjunction with the project work plans. The project work plans will commonly provide the following information:

- Sample collection objectives
- Numbers, types and locations of samples to be collected
- Any additional sample labeling requirements or procedures beyond those covered in this SOP, as necessary.

### 5.1 Sample Labeling

5.1.1 Document all the information necessary on the sample label and ensure that the label is physically attached to each respective sample. Each sample label must contain at a minimum the following information:

- Project name
- Project number
- Date and time of collection
- Sample location
- Sample identification number in accordance with SOP-05
- Collector's name
- Preservative used (if any).

Additional information may also be required per the project work plans and must accordingly be included on all sample labels.

5.1.2 Indelible ink should be used in filling out all sample labels.

5.1.3 Ensure that each sample collected has a sample label.

5.1.4 Ensure that the information documented on the sample label corresponds with the information documented on the Sample Collection Log, Sampling Information Form for groundwater samples and Chain-of-Custody Record.

## 6.0 REQUIRED FORMS

6.1 Soil Sample Collection Field Sheet

6.2 Groundwater Sampling Form

6.3 Chain-of-Custody Record

6.4 Field Log Book

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) establishes guidelines and procedures for sample numbering. Sample numbering is required to identify, track and trace samples from the time of collection until the time of disposal. Additional specific procedures and requirements will be provided in the project work plans.

## **2.0 REFERENCES**

2.1 EPA, September 1987, Compendium of Superfund Field Operations Methods, EPA 540/P-87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidance for Conducting Remedial Investigation and Feasibility Studies Under CERCLA, Interim Final OSWER Directive 9355.3-01.

## **3.0 RESPONSIBILITIES**

### 3.0 Responsibilities

3.1 The Field Team Leader is responsible for ensuring that all sample collection and numbering activities are conducted and documented in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Quality Control System Manager (QCSM) is responsible for periodic review of field generated documentation associated with this SOP. The QCSM is also responsible for implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to sample numbering requirements, issuing nonconformances, etc.) if problems occur.

3.3 The Field Team Lead(s) assigned to sampling and sample numbering activities are responsible for completing their tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from the procedures to the Field Team Leader.

## **4.0 DEFINITIONS/MATERIALS**

### 4.1 Sample Number

A sample number is a unique alphanumeric identification assigned to each and all physical samples collected as part of any given project.

## **5.0 PROCEDURE**

This section contains the procedures involved with sample numbering. Sample numbering is required to provide a means by which samples can be identified, tracked and traced from the time of collection until the time of disposal. The details within this SOP should be used in conjunction with project work plans. The project work plans will generally provide the following information:

- Sample collection objectives
- Numbers, types, and locations of samples to be collected

- Project-specific character string to be used for the sample numbering
- Person responsible for issuing sample numbers to field personnel conducting sampling activities

Any additional sample numbering requirements or procedures beyond those covered in this SOP, as necessary.

### 5.1 Sample Numbering

5.1.1 The alphanumeric character string (AANNNN vs. AAANNNNN) will be determined on a project-specific basis and stated in the project work plans. The sample numbers should be as simple and preferably as short as possible; however, they should also be compatible with the laboratory analytical tracking system and the data management system to be used for the project sample data.

5.1.2 A unique sample number will be assigned in the field to each sample to be submitted for analysis.

5.1.3 The sample numbers will be assigned sequentially as the samples are collected. For example:

- Borings: 606-1-1(Date)
- Monitoring wells: MW-01 (Date)
- Private drinking wells: PDW-01 (Date)
- Matrix Spike/Matrix Spike Duplicate: 606-1-1-MS,MSD (Date)
- Duplicate: 601-1-10 (Date)

Both environmental (soil and groundwater) and QC samples will be assigned sequential sample numbers with the same prefix so that the laboratory will be unable to distinguish between the QC and non-QC samples.

5.1.4 The sample number will be recorded, using indelible ink, directly on the sample label attached to each sample per SOP-04.

5.1.5 The sample number must also be recorded on the Sample Collection Log, Sampling Information Form for groundwater samples, and Chain-of-Custody Record.

5.1.6 It is recommended that one person (either the Field Team Lead or other designee) be responsible for issuing sample numbers to field sampling personnel and ensuring that the sample sequence numbers are applied to samples in the sequence in which they are collected.

5.1.7 It is also recommended the field supervisor or designee be responsible for keeping a master sample log listing the sample numbers and a brief description of the samples collected.

## 6.0 REQUIRED FORMS

6.1 Soil Sample Collection Field Sheet

6.2 Chain of Custody Record

6.3 Field Log Book

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) establishes guidelines and procedures for on-site sample storage. On-site sample storage may be required for samples collected during a given project. Additional on-site sample storage procedures and requirements will be provided in the project work plans.

## **2.0 REFERENCES**

2.1 EPA, September 1987, Compendium of Superfund Field Operations Methods, EPA 540/P-87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidance for Conducting Remedial Investigation and Feasibility Studies Under CERCLA, Interim Final OSWER Directive 9355.3-01.

## **3.0 RESPONSIBILITIES**

3.1 The Field Team Leader is responsible for ensuring that all on-site sample storage activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Quality Control System Manager (QCSM) is responsible for periodic review of field generated documentation associated with this SOP. The QCSM is also responsible for Implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to sample storage requirements, issuing nonconformance, etc.) if problems occur.

3.3 The Field Team Lead(s) assigned to sample storage activities are responsible for completing their tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from procedures to the Field Team Leader.

## **4.0 DEFINITIONS/MATERIALS**

### **4.1 Field sample**

A sample that has been collected at a project site, during the execution phase of the project, and for the purposes of the project, as defined in the project work plans.

### **4.2 On-site**

For purposes of this SOP, “on-site” is defined as any area within the project site.

### **4.3 On-site sample storage**

For purposes of this SOP, “on-site sample storage” applies to samples stored within the project site for a temporary period of time. Typically, samples may be stored on-site if they are in transit between the project site and a designated laboratory.

## **5.0 PROCEDURE**

This section contains the requirements pertaining to on-site sample storage. Proper storage is essential to maintain the quality and integrity of samples collected during a field project. The details within this SOP should be used in conjunction with project work plans. At a minimum, the project work plans will provide the following information:

- Sample collection objectives
- Numbers, types and locations of samples to be collected

Any additional on-site sample storage requirements or procedures beyond those covered in this SOP, as necessary.

### 5.1 On-Site Sample Storage Requirements

5.1.1 Samples of all types of media may need to be stored on-site. The manner in which these samples are stored will be appropriate for individual samples or each sample type.

5.1.2 Samples collected for chemical analysis are typically required to be stored at approximately 4 Centigrade (C). Therefore, such samples should either be preserved in a “cooler” using water ice, and/or in a “Sample-only” refrigerator until received by the assigned laboratory. If a refrigerator is used to store samples at the project site, this refrigerator will be dedicated for the sole use of samples; no food, drinks or other personal items will be allowed in this refrigerator.

5.1.3 Samples that do not require refrigeration (e.g. air samples and samples for geotechnical or radionuclide analysis) should be stored on-site in a designated, marked area.

5.1.4 Samples that are stored on-site must be stored in appropriate containers per the project specific work plans and be maintained under custody per SOP-01.

5.1.5 Samples that are stored on-site must not be stored in a manner in which they may threaten the integrity of other samples in the holding location,

5.1.6 All samples that are stored on-site must be labeled per SOP-04, numbered per SOP-05, and appropriately handled per SOP-03.

5.1.7 It is recommended the Field Team Lead or other designee be responsible for maintaining a master sample log listing sample numbers and a brief description of samples collected. The master log should be reviewed on a daily basis for samples that are under storage on site. The samples should then be appropriately shipped, following procedures per SOP-03, to ensure that holding time are not missed.

5.1.8 Samples that are not shipped to the assigned laboratory should be disposed of in a timely manner following appropriate disposal practices for the media from which the samples were initially obtained.

## 6.0 REQUIRED FORMS

### 6.1 Field Log Book

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) establishes guidelines and procedures for soil sampling. Proper collection procedures are necessary to assure the quality and integrity of all subsurface soil samples.

## **2.0 REFERENCES**

- 2.1 Environmental Protection Agency. Soil Sampling Operating Procedure. 21 August 2014.
- 2.2 Accutest Laboratories. Method 5035A Terracore / Field Kit Guidelines for Volatile Organic Analysis.
- 2.3 En Novative Technologies, Inc. Recommended Use of the Terra Core.
- 2.4 En Novative Technologies, Inc. Disposable EnCore Sampler Sampling Procedures.
- 2.5 En Novative Technologies, Inc. SW846 Method 5035 Field Sampling Guide – EnCore Sampling.

## **3.0 RESPONSIBILITIES**

- 3.1 The Field Team Leader is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).
- 3.2 The Quality Control System Manager (QCSM) is responsible for periodic review of field generated documentation associated with this SOP. The QCSM is also responsible for implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to QC sampling requirements, issuing nonconformances, etc.) if problems occur.
- 3.3 The Field Team Lead(s) assigned to soil sampling activities are responsible for completing their tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from procedures to the Field Team Leader or QCSM as appropriate.

## **4.0 DEFINITIONS/MATERIALS**

### **4.1 Borehole**

Any hole drilled into the subsurface for the purpose of identifying lithology, collecting soil samples, and/or installing monitoring wells.

### **4.2 EnCore Sampler**

An inert polymeric container designed to hold approximately 5 or 10 grams of samples that can be sealed to prevent loss of volatile organic constituents.

#### 4.3 Terra Core® Sampler

A disposable transfer tool used to deliver an approximate 5 gram sample into a 40 mL VOA vial for in-field preservation.

### 5.0 PROCEDURE

This section contains both the responsibilities and procedures involved with soil sampling. Proper subsurface soil sampling procedures are necessary to insure the quality and integrity of the samples. The details within this SOP should be used in conjunction with project work plan. The project work plans will generally provide the following information:

- Sample collection objectives
- Locations of soil borings and target horizons or depths of soil samples to be collected
- Numbers and volumes of samples to be collected
- Types of chemical analyses to be conducted for the samples
- Specific quality control (QC) procedures and sampling required

Soil samples can be collected by multiple methods, including hand augering (digging), and test pit/backhoe methods. Any additional soil sampling requirements or procedures beyond those covered in this SOP should be included in the project work plan, as necessary.

#### 5.1 Soil Sampling

5.1.1 Calibrate all field analytical and health and safety monitoring equipment according to the instrument manufacturer's specifications. Calibration results will be recorded on the appropriate form(s) as specified by the project-specific work plans. Instruments that cannot be calibrated according to the manufacturer's specifications will be removed from service and tagged.

5.1.2 Wear the appropriate personal protective equipment as specified in the project work plans and the applicable drilling method SOP. Personnel protection will typically include a hard hat, safety glasses, gloves, steel-toed boots, hearing protection, and coveralls.

5.1.3 Between each sampling location and prior to each sampling run, decontaminate the sampler, sleeves, and other sampling equipment as described in SOP-12. Wear new, clean gloves while handling sample containers and sampling devices. Change out gloves at each sampling location, or each time a new sample is to be collected to avoid cross-contamination.

5.1.6 Soil samples will be collected from the appropriate interval and location.

- Extract the sample using a stainless steel or plastic spoon or trowel and place in the appropriate sampling container.
- Samples destined for both laboratory and XRF analysis will be homogenized and split between the two sample containers.
- Wipe the sample containers with a clean paper towel to remove any residual soil from the sample container surface.
- Appropriately label and number each sample to be submitted for analysis per SOP-04 and 05, respectively. The label will be filled out using waterproof ink and will contain, at a minimum, the following information:
  - Project number

- Boring number
- Sample number
- Sample depth
- Date and time of sample collection
- Parameters for analysis
- Sampler's initials.

5.1.7 Appropriately preserve, package, handle, and ship the sample in accordance with the procedures outlined in SOP-03 and the project work plans. The samples shall also be maintained under custody per SOP-01. Samples stored on-site will be subject to the provisions of SOP-06.

5.1.8 Repeat this sampling procedure at the intervals specified in the project work plans until the bottom of the borehole is reached and/or last sample collected.

## **6.0 REQUIRED FORMS**

6.1 Soil Sample Collection Field Sheet

6.2 Field Log Book

6.3 Chain of Custody

## SW 846 METHOD 5035 FIELD SAMPLING GUIDE

### EN CORE<sup>®</sup> SAMPLER COLLECTION FOR LOW LEVEL ANALYSES ( $\geq 1$ UG/KG)

When sampling for low level analyses, a high level sample also needs to be collected.

#### EN CORE<sup>®</sup> SAMPLING

---

##### Each sample point requires

- Two 5g samplers.
- One 25g sampler or one 5g sampler for screening and/or high level analysis.  
(The sampler size used will be dependent on who is doing the sampling and who is doing the laboratory analysis).
- One dry weight cup.
- One T-handle.
- Paper toweling.

##### Procedure-Sampling

1. Remove sampler and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.
4. Push cap on with a twisting motion to attach cap.
5. Fill out label and attach to sampler.
6. Repeat procedure for the other two samplers.
7. Collect dry weight sample-fill container.
8. Store samplers at 4 degrees Celsius.
9. Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.



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## SW 846 METHOD 5035 FIELD SAMPLING GUIDE

### EN CORE<sup>®</sup> SAMPLER COLLECTION FOR HIGH LEVEL ANALYSES ( $\geq 200$ UG/KG)

#### EN CORE<sup>®</sup> SAMPLING

---

##### Each sample point requires

- One 25g sampler or one 5g sampler (The sampler size used will be dependent on who is doing the sampling and who is doing the laboratory analysis).
- One dry weight cup.
- One T-handle.
- Paper toweling.

##### Procedure-Sampling

1. Remove sampler and cap from package and attach T-handle to sampler body.
2. Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
3. Use paper toweling to quickly wipe the sampler head so that the cap can be tightly attached.
4. Push cap on with a twisting motion to attach cap.
5. Fill out label and attach to sampler.
6. Collect dry weight sample-fill container.
7. Store samplers at 4 degrees Celsius.
8. Ship sample containers with plenty of ice to the laboratory within 40 hours of collection.



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## SW 846 METHOD 5035 FIELD SAMPLING GUIDE

### ACID PRESERVATION SAMPLING FOR LOW LEVEL ANALYSES ( $\geq 1$ ug/kg)

#### ACID PRESERVATION SAMPLING

##### Each sample point requires

- One 40mL VOA vial with acid preservative (for field testing of soil pH).
- Two pre-weighed 40mL VOA vials with acid preservative and stir bar (for lab analysis).
- Two pre-weighed 40mL VOA vials with water and stir bar (in case samples effervesces).
- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol (for screening sample and/or high level analysis).
- One dry weight cup.
- One 2oz jar with NaHSO<sub>4</sub> acid preservative (in case additional acid is needed due to high soil pH).
- One scoop capable to deliver about one gram of solid sodium bisulfate.
- pH paper.
- Weighing balance that weighs to 0.01 g (field balances may not reliably weigh to 0.01g). IAETL suggests 0.1g.
- Set of balance weights used in daily balance calibration.
- Gloves for working with pre-weighed sample vials.
- Paper toweling.

##### Procedure-Field Chemistry For Testing Effervescing Capacity Of Soils

1. Place ~5g of soil into a vial that contains acid preservative and no stir bar.
2. Do not cap this vial as it may EXPLODE upon interaction with the soil.
3. Observe the sample for gas evolution (due to carbonates in the soil).
4. If vigorous or sustained gas evolution occurs: then acid preservation is not acceptable to preserve the sample.
  - In this case the samples need to be collected in the VOA vials with only water and a stir bar.
  - The vials with acid preservative CANNOT be used.
5. If a small amount or no gas evolution occurs: then acid preservation is acceptable to preserve the sample. Keep this testing vial for use in the buffering testing detailed below.
  - In this case the samples need to be collected in the VOA vials with the acid preservative and a stir bar.

##### Procedure-Field Chemistry For Testing Buffering Capacity Of Soils

1. If acid preservation is acceptable for sampling soils then the sample vial that was used in the effervescing testing can be used here for the buffering testing.
2. Cap the vial that contains ~5g of soil, acid preservative and no stir bar from step 1 in the effervescing testing.
3. Shake the vial gently to attempt to make a homogenous solution.
4. When done, open the vial and check the pH of the acid solution with the pH paper.
  - If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA vials with the acid preservative and stir bar. Since the pH was below 2, it is not necessary to add additional acid to the vials.
  - If the pH paper reads above 2, then additional acid needs to be added to the sample vial.
5. Use the jar with the solid sodium bisulfate acid and add another one gram of acid to the sample.
6. Cap the vial and shake thoroughly again.
7. When done, open the vial and check the pH of the acid solution with a new piece of pH paper.
  - If the pH paper reads below 2 then the sampling can be done in the two pre-weighed 40mL VOA vials with the acid preservative and stir bar and one extra gram of acid.
  - Make a note of the extra gram of acid needed so the same amount of extra acid can be added to the vials the lab will analyze.
  - If the pH paper reads above 2, then add another gram of acid and repeat this procedure one more time.

Now that the soil chemistry has been determined the actual sampling can occur. The procedure stated below assumes the correct vials are used based on the guidance discussed.



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## SW 846 METHOD 5035 FIELD SAMPLING GUIDE

### ACID PRESERVATION SAMPLING FOR LOW LEVEL ANALYSES ( $\geq 1$ ug/kg)

#### ACID PRESERVATION SAMPLING (CONT.)

##### Procedure-Sampling

1. Wear gloves during all handling of pre-weighed vials.
2. Quickly collect a 5g sample using a cut off plastic syringe or other coring device designed to deliver 5g of soil from a freshly exposed surface of soil.
3. Carefully wipe exterior of sample collection device with clean paper toweling.
4. Quickly transfer to the appropriate VOA vial, extruding with caution so that the solution does not splash out of the vial.
5. Add more acid if necessary (this is based on the buffering testing discussed in the previous section).
6. Use the paper toweling and quickly remove any soil off of the vial threads.
7. Cap vial and weigh the jar to the nearest 0.01 g (IAETL suggests nearest 0.1g).
8. Record exact weight on sample label.
9. Repeat sampling procedure for the duplicate VOA vial.
10. Weigh the vial with methanol preservative in it to 0.01g. If the weight of the vial with methanol varies by more than 0.01 g from the original weight recorded on the vial-discard the vial. If the weight is within tolerance it can be used for soil preservation below. (IAETL suggests weighing to the nearest 0.1g)
11. Tare the empty jar or the jar that contains the methanol preservative.
12. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size is dependent on who is doing the sampling and who is doing the laboratory analysis.
13. Carefully wipe the exterior of the collection device with clean paper toweling.
14. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol be careful not to splash the methanol outside of the vial. Again, the type of jar received is dependent on who is doing the laboratory analysis.
15. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided using only one vial of methanol preservative per sample jar.
16. Use the paper toweling and remove any soil off of the vial threads and cap the jar.
17. Weigh the jar with the soil in it to 0.01g and record the weight on the sample label. (IAETL suggests weighing to the nearest 0.1g).
18. Collect dry weight sample-fill container.
19. Store samples at 4 degrees Celsius.
20. Ship sample containers with plenty of ice and per DOT regulations (CORROSIVE, FLAMMABLE LIQUID, POISON) to the laboratory.



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## SW 846 METHOD 5035 FIELD SAMPLING GUIDE

### METHANOL PRESERVATION SAMPLING FOR HIGH LEVEL ANALYSES ( $\geq 200$ ug/kg)

## METHANOL PRESERVATION SAMPLING

#### Each sample point requires

- One pre-weighed jar that contains methanol or a pre-weighed empty jar accompanied with a pre-weighed vial that contains methanol.
- One dry weight cup.
- Weighing balance that accurately weighs to 0.01g (field balances may not reliably weigh to 0.01g). IAETL suggests 0.1g.
- Set of balance weights used in daily balance calibration.
- Gloves for working with pre-weighed sample vials.
- Paper toweling.

#### Procedure-Sampling

1. Wear gloves during all handling of pre-weighed vials.
2. Weigh the vial with methanol preservative in it to 0.01g. If the weight of the vial with methanol varies by more than 0.01 g from the original weight recorded on the vial-discard the vial. If the weight is within tolerance it can be used for soil preservation/collection below (IAETL suggests weighing to the nearest 0.1g).
3. Tare the empty jar or the jar that contains the methanol preservative.
4. Quickly collect a 25g or 5g sample using a cut off plastic syringe or other coring device designed to deliver 25g or 5g of soil from a freshly exposed surface of soil. The 25g or 5g size used is dependent on who is doing the sampling and who is doing the laboratory analysis.
5. Carefully wipe the exterior of the collection device with clean paper toweling.
6. Quickly transfer the soil to an empty jar or a jar that contains methanol. If extruding into a jar that contains methanol be careful not to splash the methanol outside of the vial. Again, the type of jar used is dependent on who is doing the laboratory analysis.
7. If the jar used to collect the soil plug was empty before the soil was added, immediately preserve with the methanol provided-using only one vial of methanol preservative per sample jar.
8. Using the paper toweling-remove any soil off of the vial threads and cap the jar.
9. Weigh the jar with the soil in it to 0.01g and record the weight on the sample label. (IAETL suggest weighing to the nearest 0.1g).
10. Collect dry weight sample-fill container.
11. Store samples at 4 degrees Celsius
12. Ship sample containers with plenty of ice and per DOT regulations (CORROSIVE, FLAMMABLE LIQUID, POISON) to the laboratory.



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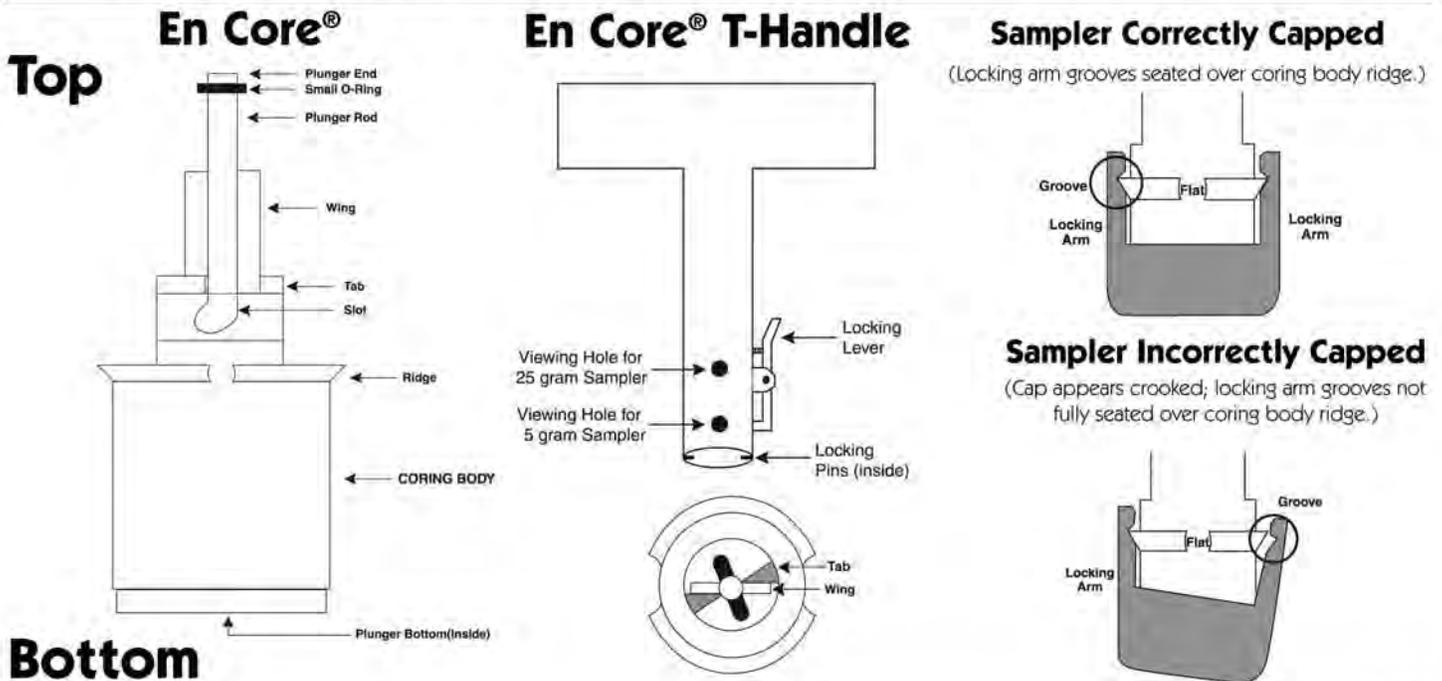
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# Disposable En Core® Sampler Sampling Procedures

## Using The En Core® T-Handle

### NOTE:

1. En Core® Sampler is a SINGLE USE device. It cannot be cleaned and/or reused.
2. En Core® Sampler is designed to store soil. Do not use En Core Sampler to store solvent or free product!
3. En Core® Sampler must be used with En Core® T-Handle and/or En Core® Extrusion Tool exclusively. (These items are sold separately.)



### BEFORE TAKING SAMPLE:

1. Hold **coring body** and push **plunger rod** down until **small o-ring** rests against **tabs**. This will assure that plunger moves freely.
2. Depress **locking lever** on En Core T-Handle. Place coring body, **plunger end first**, into open end of T-Handle, **aligning the (2) slots on the coring body with the (2) locking pins in the T-Handle**. Twist coring body clockwise to lock pins in slots. Check to ensure Sampler is locked in place. Sampler is ready for use.

### TAKING SAMPLE:

3. Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body (ensure that plunger bottom is in position). Using T-Handle, push Sampler into soil until coring body is completely full. When full, small o-ring will be centered in T-Handle **viewing hole**. Remove Sampler from soil. Wipe excess soil from coring body exterior.

4. Cap coring body while it is still on T-handle. **Push cap over flat area of ridge and twist** to lock cap in place. **CAP MUST BE SEATED TO SEAL SAMPLER (see diagram)**.

### PREPARING SAMPLER FOR SHIPMENT:

5. Remove the capped Sampler by depressing locking lever on T-Handle while twisting and pulling Sampler from T-Handle.
6. Lock plunger by rotating extended plunger rod fully counter-clockwise until **wings** rest firmly against tabs (see plunger diagram).
7. Fill in sample description on the back of the En Core Sampler bag.
8. Return full En Core Sampler to zipper bag. Seal bag and put on ice.



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# Disposable En Core® Sampler

## **EXTRUSION PROCEDURES**

### USING THE En Core® EXTRUSION TOOL

**CAUTION!** Always use the Extrusion Tool to extrude soil from the En Core Sampler. If the Extrusion Tool is not used, the Sampler may fragment, causing injury.

1. To attach En Core Sampler to En Core Extrusion Tool: Depress locking lever on Extrusion Tool and place Sampler, plunger end first, into open end of Extrusion Tool, aligning slots on coring body with pins in Extrusion Tool. Turn coring body clockwise until it locks into place. Release locking lever.
2. Rotate and gently push Extrusion Tool plunger knob clockwise until plunger slides over wings of coring body. (When properly positioned plunger will not rotate further.)
3. Hold Extrusion Tool with capped Sampler pointed upward so soil does not fall out when cap is removed. Remove cap from Sampler by rotating cap until locking arms are aligned with the flat area of ridge and pull cap off. To release soil core push down on plunger knob of En Core Extrusion Tool. Remove and properly dispose of En Core Sampler.

## **Warranty and Disclaimers**

**IMPORTANT:** FAILURE TO USE THE EN CORE® SAMPLER IN COMPLIANCE WITH THE WRITTEN INSTRUCTIONS PROVIDED HEREIN VOIDS ALL EXPRESS AND IMPLIED WARRANTIES, INCLUDING WARRANTY OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

**PRINCIPLE OF USE.** The En Core Sampler Cartridge System is a volumetric sampling system designed to collect, store and deliver a soil sample. The En Core Sampler comes in two sizes for sample volumes of approximately 25 or 5 grams. There are four components: the cartridge with a movable plunger; a cap with two locking arms; a T-handle (purchased separately); and an extrusion handle (purchased separately). NOTE: The En Core Sampler is designed to store soil. It is not designed to store solvent or free product.

The soil is stored in a sealed headspace-free state. The seals are achieved by three special Viton® \* o-rings, two located on the plunger and one on the cap of the Sampler. At no time and under no condition should these o-rings be removed or disturbed.

**QUALITY CONTROL.** The cartridge is sealed in an airtight package to prevent contamination prior to use. Due to the stringent quality control requirements associated with the use of this system, the disposable cartridge is designed to be used only once.

**WARRANTY.** En Novative Technologies warrants that the En Core Sampler shall perform consistent with the research conducted under En Novative Technologies' approval, within thirty (30) days from the date of delivery, provided that the Customer gives En Novative Technologies prompt notice of any defect or failure to perform and satisfactorily proof thereof. THIS WARRANTY DOES NOT APPLY TO THE FOLLOWING, ASSOLELY DETERMINED BY EN NOVATIVE TECHNOLOGIES: (a) Damage caused by accident, abuse, mishandling or dropping; (b) Samplers that have been opened, taken apart or mishandled; (c) Samplers not used in accordance with the directions; and (d) Damages exceeding the cost of the sampler. Seller warrants that all En Core Samplers

shall be free from defects in title. THE FORE-GOING WARRANTIES ARE IN LIEU OF ALL OTHER WARRANTIES, WHETHER ORAL, WRITTEN, EXPRESSED, IMPLIED OR STATUTORY, INCLUDING ANY INFORMATION PROVIDED BY SALES REPRESENTATIVES OR IN MARKETING LITERATURE. IMPLIED WARRANTIES OF FITNESS AND MERCHANTABILITY SHALL NOT APPLY. En Novative Technologies' warranty obligations and Customer's remedies, except as to title, are solely and exclusively as stated herein.

**LIMITATION OF LIABILITY.** IN NO EVENT SHALL EN NOVATIVE TECHNOLOGIES BE LIABLE FOR ANTICIPATED PROFITS, INCIDENTAL, SPECIAL OR CONSEQUENTIAL DAMAGES, INCLUDING, BUT NOT LIMITED TO, DAMAGES FOR LOSS OF REVENUE, DOWN TIME, REMEDIATION ACTIVITIES, REMOBILIZATION OR RESAMPLING, COST OF CAPITAL, SERVICE INTERRUPTION OR FAILURE OF SUPPLY, LIABILITY OF CUSTOMER TO A THIRD PARTY, OR FOR LABOR, OVERHEAD, TRANSPORTATION, SUBSTITUTE SUPPLY SOURCES OR ANY OTHER EXPENSE, DAMAGE OR LOSS, INCLUDING PERSONAL INJURY OR PROPERTY DAMAGE. En Novative Technologies' liability on any claim of any kind shall be replacement of the En Core Sampler or refund of the purchase price. En Novative Technologies shall not be liable for penalties of any description whatsoever. In the event the En Core Sampler will be utilized by Customer on behalf of a third party, such third party shall not occupy the position of a third-party beneficiary of the obligation or warranty provided by En Novative Technologies, and no such third party shall have the right to enforce same. All claims must be brought within one (1) year of shipment, regardless of their nature.

The En Core™ Sampler is covered by One or More of the Following U.S. Patents: 5,343,771; 5,505,098; 5,517,868; 5,522,271. Other U.S. and Foreign Patents Pending.

\* Viton® is a registered trademark of DuPont Dow Elastomers.



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## Method 5035A Terracore/Field Kit Guidelines For Volatile Organic Analysis

### LOW LEVEL

Use low level sampling whenever the targeted 8260 reporting limits are in the 1-5 ug/kg range.

#### For EACH sample collected use

#### 1 Terra Core + 1 10ml Methanol preserved bottle + 2 5ml DI water bottles)

- Push the Terra Core into the soil until the chamber is filled.  
Using the plunger, deliver the soil into one of the three vials. Repeat the procedure 2 more times, adding 5 grams of soil to each of the three vials provided in the kit.
- A separate bottle of soil must accompany each field preserved sample. This can be a % solids jar or a corresponding sample jar being used for additional analysis on the same sample.
  - DO NOT affix labels to, or write on, field preserved soil samples.  
Use tags with metal twists, and attach to sample bottles.
  - **Maintain samples on ice and return the samples to the laboratory ASAP.**  
**Samples must be preserved by the lab within 48 hrs of collection.**  
**Field preserved DI water vials must be received by the lab and frozen within 48 hrs after sampling.**

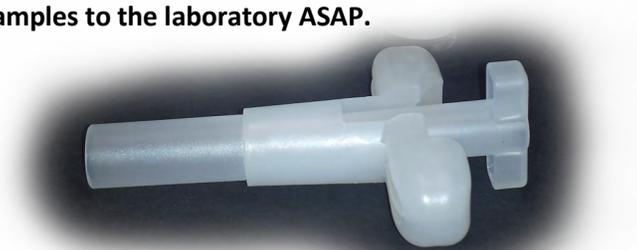
### MEDIUM LEVEL

Use medium level sampling whenever the targeted 8260 reporting limits are in the 50-250 ug/kg range.

#### For EACH sample collected use

#### 1 Terra Core and 1 10ml Methanol preserved bottle

- Push the Terra Core into the soil until the chamber is filled.  
Using the plunger, deliver 5 grams of the soil into the vial of methanol provided in the kit.
- A separate bottle of soil must accompany each field preserved sample.  
This can be a % solids jar or a corresponding sample jar being used for additional analysis on the same sample.
- DO NOT affix labels to, or write on, field preserved soil samples.  
Use tags with metal twists, and attach to sample bottles.
- **Maintain samples on ice and return the samples to the laboratory ASAP.**



# Recommended Use Of The Terra Core®



**NOTE:** The Terra Core® Sampler is a single use device. It cannot be cleaned and/or reused.



## Step 1

Have ready a 40ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core® into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 or 10 grams of soil.



## Step 2

Wipe all soil or debris from the outside of the Terra Core® sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.



## Step 3

Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial. **Note:** When capping the 40ml VOA vial, be sure to remove any soil or debris from the top and/or threads of the vial.

## SOP-08

**COMPOSITE SAMPLE PREPARATION  
STANDARD OPERATING PROCEDURE****1.0 PURPOSE**

This Standard Quality Operating Procedure (SOP) describes the requirements for compositing techniques. Composite samples, regardless of the media, consist of two or more subsamples taken from a specific media and site at different depth intervals. The subsamples are collected and mixed. A single average sample is taken from the mixture. Composite samples will be collected from the base of the excavation and for waste characterization. Composite samples are useful in estimating the overall contamination properties of a specific site. They are less expensive than non-composite samples because one sample for analysis represents many subsample locations. Composite samples do not provide detailed information of contamination variability as a function of the location.

**2.0 REFERENCES**

2.1 None.

**3.0 DEFINITIONS/MATERIALS**

The equipment required to obtain duplicate and/or split samples is identical to that for primary media sampling.

**4.0 RESPONSIBILITIES**

4.1 The Field Team Leader will ensure that sampling efforts are conducted in accordance with this procedure and other SOPs pertaining to specific media sampling.

4.2 The Quality Assurance System Manager (QCSM) is responsible for ensuring that this procedure is correctly implemented and that the quantity and quality of composite samples meet the requirements of the Sampling and Analysis Plan

4.3 The Field Team Lead(s) assigned to this task are responsible for ensuring that field personnel collect and prepare composite samples in accordance with this procedure.

**5.0 PROCEDURES****5.1 Preparation**

Site preparation for the purpose of compositing samples is not different from that required for any of the media/waste sampling activities.

**5.2 Soil Compositing**

The following steps must be followed when compositing soil samples:

- Determine where composite sample(s) will be obtained as indicated in the site-specific sampling plan.
- Collect a minimum of three equal-volume samples from the specified sample location. The volume of each sample must be at least the amount required for a single sample.
- Place the samples on an appropriate mixing tray. Thoroughly homogenize the pooled samples using the appropriate equipment.
- Transfer subsamples of the composited sample into the appropriate sample containers. Seal, decontaminate, and label sample containers. Use the same care in handling these samples as that used for other samples from the site.
- Decontaminate sampling equipment.

## **6.0 REQUIRED FORMS**

6.1 Field Log Book

6.2 Soil Sample Collection Field Sheet

6.3 Chain of Custody

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) establishes the methods and responsibilities associated with the calibration, control, and maintenance of measuring and test equipment (M&TE). It applies to all tools, gauges, instruments, and other test equipment where the manufacturer requires or recommends equipment accuracy to be checked periodically. In the case of commercial devices such as rulers, tape measures, and levels calibration controls will not be required.

## **2.0 REFERENCES**

2.1 None

## **3.0 RESPONSIBILITIES**

3.1 The Quality Assurance System Manager (QCSM) or his/her designee is responsible for monitoring the effective implementation of this SOP and/or the M&TE manufacturer's recommendations.

3.2 The Field Team Leader and Field Team Lead(s) are responsible for the selection of M&TE to be used in the field activity and to assure it is of the proper type, range, accuracy and tolerance required to meet project objectives. Additionally, he/she is responsible for storage and protection of M&TE.

3.3 The field personnel performing tests are responsible for assuring that all M&TE is properly calibrated prior to and during use, and for documenting the calibration or deficiencies of equipment.

## **4.0 DEFINITIONS**

### **4.1 M&TE**

Measuring and test equipment used to obtain data during the performance of tests or inspections.

### **4.2 Calibration**

The comparison of a measurement standard or instrument of a known accuracy with another standard or instrument to detect, correlate, report, or eliminate by adjustment, any variation in the accuracy of the items being compared within allowable deviations.

### **4.3 Reference Standard**

An item of known and verifiable value which is used to check or establish the basis for tests or inspections.

## **5.0 Procedure**

### **5.1 Equipment Identification and Control**

5.1.1 M&TE that requires calibration will be uniquely identified by the manufacturer's serial number, or other suitable assigned number. If this should prove to be impractical, an identification label will be affixed using materials and methods which provide a clear and legible

identification and do not detrimentally affect the function or service life of the M&TE. This identification will be replaced as needed to provide clear identification of the M&TE.

5.1.2 All M&TE and reference standards shall be stored between uses in a manner that will minimize damage or deterioration.

## 5.2 Calibration

5.2.1 Written and approved procedures will be used for calibration of M&TE. Calibration procedures that have been previously established and approved by the M&TE manufacturer or a nationally recognized authority (i.e., ASTM, EPA) will be used when available. If no preexisting procedure is available, procedures will be developed by qualified personnel familiar with the M&TE and approved by the PM and QCSM. Development of procedures will take into consideration the intended use and objective of the resulting data, equipment characteristics, required accuracy and precision of data, location of examination, effects of climate or any other parameter which would adversely influence the calibration. The procedures will include, as applicable:

- Name/type of equipment to be calibrated
- Reference standards to be used
- Calibration method and sequential actions
- Acceptance criteria
- Frequency of calibrations/checks
- Data recording form/format
- Data processing methodology
- Any special instructions
- Operator training and qualification requirements.

5.2.2 Field M&TE will be calibrated prior to use. Calibrations of M&TE will be performed by trained and qualified personnel, approved external agencies or by the equipment manufacturer.

5.2.3 The following types of calibrations and checks will be performed by qualified personnel:

- Periodic calibrations - which are performed at prescribed intervals established for the M&TE to assure that the equipment is operating within its designed range and accuracy. These are usually performed by outside agencies or the M&TE manufacturer. A calibration certificate will be provided documenting the operational and functional acceptance of the M&TE.
- Specific calibrations - which are performed for specific measurements or tests and varies from instrument to instrument and from procedure to procedure. Specific calibrations are performed prior to start of each work shift.

## 5.3 Calibration Frequency

5.3.1 M&TE will be calibrated at prescribed intervals and before each specific use. The frequency of periodic calibrations will be based on manufacturer's recommendations, national standards of practice, equipment type and characteristics, and past experience.

5.3.2 Scheduled calibrations of M&TE does not relieve the user of the responsibility for selecting the appropriate and properly functioning equipment.

5.3.3 In the event that the calibration has expired, the M&TE will be removed from service and tagged as “out-of-service” to prevent inadvertent use until it has been appropriately recalibrated.

#### 5.4 Reference Standards and Equipment

5.4.1 Calibration reference standards and equipment will have known relationships to the National Institute of Standards and Technology (NIST) or other nationally recognized standards. If a national standard does not exist, the basis for calibration will be fully documented by the Project Manager and approved by the QCSM.

5.4.2 Physical and chemical standards will have certifications traceable to NIST, EPA or other recognized agencies. Standards that are repackaged or split will also have traceable lot or batch numbers transferred onto the new container.

5.4.3 It is the responsibility of the user to select, verify and use the correct standard in accordance with an approved procedure or established practice.

#### 5.5 Calibration Failure

5.5.1 Each individual user of M&TE is responsible for checking the calibration status of equipment to be used and confirming the acceptable calibration status prior to use. Equipment for which the periodic calibration period has expired, equipment that fails calibration, or equipment that becomes inoperable during use will be removed from service and tagged as out-of-service.

5.5.2 Out-of-service M&TE will be segregated from operational M&TE when practical. The specific reason for removal from service and the date of removal will also be stated on the out-of service tag. The M&TE will then be repaired and/or recalibrated by the appropriate vendor or manufacturer as deemed necessary by the PM. M&TE that cannot be repaired will be replaced, as necessary, to provide support to the project. Any M&TE consistently found to be out-of-calibration will be replaced.

5.5.3 Results of activities performed using equipment that has failed recalibration will be evaluated by the Project Manager and QCSM. If the activity results are adversely affected, the results of the evaluation will be documented as a nonconformance.

#### 5.6 Calibration Documentation

5.6.1 Specific calibration records will be prepared and documented for each calibrated M&TE used. Periodic calibration certificates will be maintained and available for review at the field office. Calibration data will be recorded on the Test Equipment List and Calibration Log form or other suitable form. The Project Manager will be responsible for reviewing the calibration data for appropriateness, accuracy, readability, and completeness.

5.6.2 Calibration records will include, as applicable, the following information:

- Equipment identification number
- Calibration procedure used
- Date/time of calibration
- Time of calibration checks (if required)
- Identification of reference standard(s) used
- Applicable responses or readings of calibration
- Name of individual performing calibration
- Item(s) that are being tested or inspected.

### 5.7 Preventive Maintenance

5.7.1 Preventive maintenance of M&TE will be performed in accordance with manufacturer's recommendation to maintain proper M&TE performance, minimize equipment failure and to increase measurement reliability.

## **6.0 REQUIRED FORMS**

6.1 Instrument Calibration Log

6.2 Field Log Book

## SOP-10

**FIELD INSTRUMENT QA/QC  
STANDARD OPERATING PROCEDURE****1.0 PURPOSE**

The purpose of this Standard Operating Procedure (SOP) is to define field requirements for quality assurance/quality control (QA/QC), for equipment and instrument calibration, inspection, and maintenance. Instruments and equipment used to gather, generate, or measure environmental data must be calibrated to ensure that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Equipment, instruments, tools, gauges, and other items requiring preventive maintenance must be serviced according to the manufacturer's specifications. Raw data from the field measurements and sample collection activities must be recorded in the appropriate logbook or field form, and standard reporting units must be used for comparability and consistency.

**2.0 REFERENCES**

2.1 EPA, September 1987, EPA Compendium of Superfund Field Operations Methods, EPA 540/P-87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidelines for Conducting Remedial Investigation and Feasibility Studies under CERCLA, Interim Final OSWER Directive 9355.3-01.

**3.0 RESPONSIBILITIES**

3.1 The Field Team Leader is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Quality Assurance System Manager (QCSM) has the responsibility for periodic review of procedures and documentation associated with the calibration of field instrumentation. If perceived variances occur, the QCSM is also responsible for issuing notices of nonconformances and requesting corrective actions.

3.3 The Field Team Lead / Site Safety and Health Officer (SSHO) is responsible for ensuring that calibration is completed daily in accordance with this procedure, that equipment and instrument inspection and maintenance is conducted, that measurements are taken to the specified accuracy. The SSHO is also responsible for validation of field data by:

- Conducting routine checks during the processing of data (e.g. errors in identification codes);
- Checking the consistency with parallel data sets obtained presumably from the same population (e.g., from the same portion of the aquifer or volume of soil).

3.4 The Field Team Lead(s) are responsible for calibrating, inspecting, and maintaining instruments, taking measurements to the specified precision.

#### **4.0 DEFINITIONS/MATERIALS**

4.1 Instruments (to be calibrated, and manufacturer's operating manual)

- X-Ray Fluorescence Spectrometers

4.2 Other:

- Maintenance schedule.
- Field logbook.
- Indelible black or blue ink pens.

#### **5.0 PROCEDURE**

5.1 Equipment and Instrument Calibration

The frequency of calibration for field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate, but daily as a minimum. Calibration procedures will be completed in accordance with the equipment manual for each field instrument. A list of potential field instruments is included in Section 4.1 above. Calibration will be documented on the Equipment Calibration Form.

To ensure comparability between sample data of similar samples and sample conditions, standard solutions and material traceable to the National Institute of Standards and Technology or EPA published standards/protocols will be used to calibrate the field instruments.

5.2 Equipment and Instrument Inspection and Maintenance

5.2.1 Equipment and Instrument Inspection

Equipment to be used during field sampling will be examined to ensure that it is in proper operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to ensure that all maintenance requirements are being observed. Field notes for previous sampling trips will be reviewed so that the notations on any prior equipment problem are not overlooked and all necessary repairs to equipment have been carried out.

5.2.2 Equipment and Instrument Maintenance

Equipment, instruments, tools, gauges, and other items requiring preventive maintenance will be serviced in accordance with the manufacturer's recommendations.

Manufacturer's procedures identify the schedule for servicing critical items in order to minimize the downtime of the measurement system. It will be the responsibility of the operator to adhere to the maintenance schedule and to arrange any necessary and prompt service as required. Service to the equipment, instruments, tools, gauges, etc., will be performed by qualified personnel. In the absence of any manufacturer's recommended maintenance criteria, a maintenance procedure will be developed by the operator based upon experience and previous use of the equipment.

Logs will be established to record maintenance and service procedures and schedules. All maintenance records will be documented and traceable to the specific equipment, instruments, tools, and gauges.

### 5.3 Field Measurement Precision

- The XRF is calibrated by the supplier according to manufacturer specifications. The calibration log from the most recent calibration will be requested from the supplier and included with the paperwork for this project.
- A check standard provided by the manufacturer will be measured each day to test both the precision and accuracy of the instrument measurements. Refer to SOP-22 for additional information.

## **6.0 REQUIRED FORMS**

### 6.1 Instrument Calibration Log

### 6.2 Field Log Book

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) describes the procedures required for decontamination of field equipment. Decontamination of field equipment is necessary to ensure the quality of samples by preventing cross-contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants off site.

## **2.0 REFERENCES**

2.1 EPA, September 1987, EPA Compendium of Superfund Field Operations Methods, EPA 540/P-87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidelines for Conducting Remedial Investigation and Feasibility Studies under CERCLA, Interim Final OSWER Directive 9355.3-01.

## **3.0 RESPONSIBILITIES**

3.1 The Field Team Leader is responsible for ensuring that all equipment decontamination activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Project Chemist are responsible for ensuring that field personnel are trained in the use of this procedure and that decontamination is conducted in accordance with this procedure.

3.3 The Quality Assurance System Manager (QCSM) has the responsibility for periodic review of procedures and documentation associated with the decontamination of drilling and heavy equipment. If perceived variances occur, the QCSM is also responsible for issuing notices of nonconformances and requesting corrective actions. Additionally, he/she will perform the three phases of inspections and continuous monitoring of the decontamination activities.

3.4 The Field Team Lead(s) are responsible for verifying that this procedure is correctly implemented. The Field Team Lead may also be required to collect and document rinse water samples to provide quantitative verification that these procedures have been correctly implemented. This SOP and the project work plans should be reviewed before implementing decontamination procedures at the project field area.

## **4.0 DEFINITIONS/MATERIALS**

### **4.1 Deionized Analyte-Free Water**

Ion-free, analyte-free water produced on site or purchased from a supplier with a deionization chamber equipped with a carbon filter.

### **4.2 Potable Water**

Treated municipal water.

### **4.3 Laboratory Grade Detergent**

A standard brand of laboratory-grade detergent, such as “Alconox” or “Liquinox”

#### 4.4 Nonsampling Equipment

Nonsampling equipment includes:

- Field logbook.
- Drilling rigs, augers, drill pipe, bits, casing, and screen.
- High-pressure pump soap dispenser or steam-spray unit.
- 2- to 5-gal manual-pump sprayer (pump sprayer material must be compatible with the solution used).
- Stiff-bristle brushes.
- Gloves, goggles, boots, and other protective clothing as specified in the site-specific health and safety plan.

#### 4.5 Small Equipment

Small equipment includes:

- Groundwater monitoring probes.
- Groundwater sampling pumps.
- 5-gal plastic buckets
- Laboratory-grade detergent (phosphate free).
- Stiff-bristle brushes.
- Nalgene, or Teflon, sprayers or wash bottles or 2- to 5-gal manual-pump sprayer (pump sprayer material must be compatible with the solution used).
- Plastic sheeting.
- Disposable wipes or rags.
- Potable water.
- Appropriate decontamination solutions.
- Gloves, goggles, and other protective clothing as specified in the site-specific health and safety plan.

### **5.0 PROCEDURES**

This section contains responsibilities, requirements, and procedures for sampling equipment and well material decontamination. The decontamination is required to maintain proper quality and integrity of collected samples.

The details within this SOP should be used in conjunction with the project work plans. The project work plans will provide the following information:

- Types of equipment requiring decontamination under this SOP; specific materials to be used for the decontamination; and
- Additional decontamination requirements and procedures beyond those covered in this SOP, as necessary.

All field personnel associated with decontamination of sampling equipment or well materials must read both this SOP and the project work plans prior to implementation of related

decontamination activities. Information and requirements for the decontamination of any and all drilling and heavy equipment is provided in SOP-13

### 5.1 Decontamination Facility

If possible, sampling equipment decontamination will take place in an area designed exclusively for decontamination. This area will ideally be located within the contamination reduction zone on the project site. Well materials may be decontaminated at the facility set up for decontamination of drilling and heavy equipment (see SOP-13).

Each decontamination facility will be constructed so that the equipment, as well as all wastes generated during decontamination (e.g.: soil, rinsate, liquid spray, debris, etc.), are fully contained. In addition, chemical products used in the decontamination process must be properly containerized and labeled.

### 5.2 Decontamination of Sampling Equipment

Each piece of reusable, small or nondedicated sampling equipment will be decontaminated before mobilization to each site and before each sampling event. The standard procedure will be performed as described below.

5.2.1 Suitable personal protective equipment (specified by the project work plans) must be worn by all personnel involved with the task to reduce personal exposure.

5.2.2 Heavily caked soil and/or other material will be scraped or brushed from equipment. The scrapings will be placed into an appropriate container for disposal. Steam cleaning of equipment may be required to remove material from samplers.

5.2.3 Equipment that will not be damaged by water should be placed into a wash tub or 5 gallon bucket containing a laboratory-grade detergent solution and scrubbed with a brush or clean cloth. Rinsing will then be conducted with fresh, potable water, followed by deionized water.

5.2.4 Any equipment that may be damaged by submersion into water will be wiped clean using a sponge and detergent solution. Cleaning will be followed by wiping the equipment with deionized water.

5.2.5 Air dry the rinsed equipment. Soil organic vapor (SOV) sampling equipment should be flushed dry with bottled air of known quality and/or as per the project work plans.

5.2.6 Place decontaminated equipment on clean plastic sheeting to prevent contact with contaminated soil. If equipment is not used immediately, cover or wrap the equipment in clean plastic sheeting to minimize airborne contamination.

5.2.7 Decontamination activities shall be documented in the field logbook or other appropriate form(s), as specified by the project work plans.

### 5.3 Waste Disposal

The following steps must be followed when disposing of wastes:

- All wash water and rinse water that have come in contact with contaminated equipment are to be handled, packaged, labeled, marked, stored, and disposed of as investigation-derived waste unless other arrangements are approved in advance.
- Small quantities of decontamination solutions may be allowed to evaporate to dryness.
- If large quantities of used decontamination solutions are generated, segregate each type of waste in separate containers. This may permit the disposal of wash water and rinse water in a sanitary sewage treatment plant rather than as a hazardous waste.
- Unless required, plastic sheeting and disposable protective clothing may be treated as a solid nonhazardous waste.

## **6.0 REQUIRED FORMS**

### 6.1 Field Logbook.

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) establishes guidelines for use by field personnel in the decontamination of drilling, development, and heavy equipment. The details within this SOP are applicable as general requirements for drilling and heavy equipment decontamination, and should also be used in conjunction with project work plans.

## **2.0 REFERENCES**

2.1 EPA, September 1987, EPA Compendium of Superfund Field Operations Methods, EPA 540/P-87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidelines for Conducting Remedial Investigation and Feasibility Studies under CERCLA, Interim Final OSWER Directive 9355.3-01.

## **3.0 RESPONSIBILITIES**

3.1 The Field Team Leader has the responsibility for ensuring that the decontamination of drilling and heavy equipment is properly performed through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Quality Assurance System Manager (QCSM) has the responsibility for periodic review of procedures and documentation associated with the decontamination of drilling and heavy equipment. If perceived variances occur, the QCSM is also responsible for issuing notices of nonconformances and requesting corrective actions. Additionally, he/she will perform the three phases of inspections and continuous monitoring of the decontamination activities.

3.3 The Field Team Lead(s) assigned to drilling, development, trenching, or construction activities are responsible for ensuring that subcontractors or equipment operators properly decontaminate the drilling, development, and heavy equipment associated with those tasks. The project staff are also responsible for documenting the decontamination activities on the field logbook and/or appropriate form(s) as specified in the project work plans.

This SOP and the project work plans should be reviewed before implementing decontamination procedures at the project field area.

## **4.0 DEFINITIONS/MATERIALS**

4.1 Laboratory Grade Detergent - A standard brand of laboratory-grade detergent, such as "Alconox" or "Liquinox".

4.2 Potable Water - Water dispensed from a municipal water system.

## **5.0 PROCEDURE**

### **5.1 General**

5.1.1 This section provides requirements for the set up of a decontamination facility for drilling, development, and heavy equipment and the decontamination procedures to be followed. The project work plans will provide specific information regarding:

- Types of equipment requiring decontamination under this SOP;
- Location of the decontamination station;
- Types and/or specifications on materials to be used in the fabrication of the decontamination station; and
- Types of materials and additional details on the procedures to be used in the decontamination process.

5.1.2 All field personnel associated with either the fabrication of the decontamination station or the decontamination of drilling or heavy equipment must read both this SOP and the project work plans prior to implementation of related decontamination activities. Information and requirements for the decontamination of any and all equipment used specifically for sampling is presented in SOP-12.

## 5.2 Decontamination Facility

5.2.1 A decontamination station will be set up in an area exclusively for decontamination of drilling, well development, and/or heavy equipment. The location of the decontamination station will be specified in the project work plans. All decontamination of drilling, development, and heavy equipment will be conducted within the station.

5.2.2 At a minimum, the station will be constructed such that all rinsates, liquid spray, soil, debris, and other decontamination wastes are fully contained and may be collected for appropriate waste management and disposal. The station may be as simple as a bermed, impermeable polyethylene sheeting, of sufficient thickness, with an impermeable sump for collecting rinse water. More sophisticated designs involving self-contained metal decontamination pads in combination with bermed polyethylene sheeting may also be used, depending on project-specific requirements. These requirements along with specific equipment and construction specifications for the decontamination station will be provided in the project work plans.

## 5.3 Decontamination of Downhole Equipment

5.3.1 All downhole drilling and development equipment (including but not limited to drill pipe, drive casing, drill rods, bits, tools, bailers, etc.) will be thoroughly decontaminated before mobilization onto each site and between borings or wells at each site or as required in the project work plans. The standard procedure will be performed as described below. Decontamination will be performed in accordance with this SOP and the project work plans.

5.3.2 Appropriate personal protective equipment (as specified in the project work plans) must be worn by all personnel involved with the task to limit personal exposure.

5.3.3 Equipment caked with drill cuttings, soil, or other material will initially be scraped or brushed. The scrapings will be containerized and appropriately disposed.

5.3.4 Equipment will then be sprayed with potable water using a hot water, high pressure washer.

5.3.5 Washed equipment will then be rinsed with potable water.

5.3.6 Decontaminated downhole equipment (such as drill pipe, drive casing, bits, tools, bailers, etc.) will be placed on clean plastic sheeting to prevent contact with contaminated soil and allowed to air dry. If equipment is not used immediately, it will be covered or wrapped in plastic sheeting to minimize airborne contamination.

5.3.7 Decontamination activities will be documented by the Field Team Lead or lead geologist on the field logbook and/or appropriate form(s), as specified in the project work plans.

#### 5.4 Decontamination of Heavy Equipment

5.4.1 Heavy equipment (e.g., drill rigs, development rigs, backhoes, and other earthmoving equipment) will be decontaminated between drilling sites or inside the contaminant reduction area prior to entering and leaving an exclusion zone. Decontamination will be performed in accordance with the project work plans. The standard procedure will be performed as described below.

5.4.1.1 Appropriate personal protective equipment (as specified in the project work plans) will be worn by all personnel involved in the task, in order to limit personal exposure.

5.4.1.2 Equipment caked with drill cuttings, soil, or other material will be initially scraped or brushed. The scrapings will be containerized and appropriately disposed.

5.4.1.3 Equipment will then be sprayed with potable water using a hot water, high pressure washer.

5.4.1.4 Clean equipment will then be rinsed with potable water.

5.4.2 During the decontamination effort, fluid systems should be inspected for any leaks or problems which might potentially result in an inadvertent release at the site, thereby contributing to the volume of waste or contamination. Any identified problems should be immediately repaired and documented on the field logbook. Decontamination should then be completed before moving the equipment onto the site or exclusion zone.

5.4.3 Decontamination activities will be documented by the Field Team Lead, or lead geologist on the field logbook and/or appropriate form(s), as specified in the project work plans.

5.4.4 Between boreholes at the same site, the back-end of the drilling rigs will be washed with potable water until surfaces are visibly free of soil buildup.

## 6.0 REQUIRED FORMS

6.1 Field Logbook.

## 1.0 PURPOSE

The objective of this procedure is to define the requirements necessary for borehole and sample logging. The major objective of this procedure is to provide a uniform set of guidelines that will aid in developing consistency among sample descriptions and sample techniques. The importance of accurate, complete, clear, and concise logs cannot be overemphasized.

## 2.0 REFERENCES

2.1 American Society for Testing and Materials (ASTM), Standard Practice for Description and Identification of Soils (Visual – Manual Procedure). ASTM Designation: D-2488

2.2 ASTM, Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System). ASTM Designation: D-2487

## 3.0 RESPONSIBILITIES

Field Geologist: The Field Geologist is responsible for on-site monitoring of drilling and soil sampling operations, for recording (logging) pertinent information regarding the geologic materials penetrated during the operations, and that the well and sample numbering system is consistent with SOP-05.

## 4.0 REQUIRED EQUIPMENT

- Clipboard
- Drilling record forms
- Portable organic vapor detector
- Field book, straight edge and black or blue permanent ink
- Foot engineer's tape (weighted)
- Folding rule or tape measure
- Sand gauge
- Color chart
- Acid bottle
- Water level indicator
- Site map
- Copy of drilling contract
- Waterproof marking pen
- Sample jars or bags

### 4.1 Optional Equipment

- Hand lens
- Brunton compass
- Pocket penetrometer
- Equipment pouch
- Flagging tape
- Cooler and water bottles
- Flashlight

- Rock hammer

## 5.0 PROCEDURE FOR FILLING OUT SOIL BORING/WELL LOG

This form is intended for use in the field during the drilling, sampling, and logging process for soil borings and wells. Most of the items can be neatly and legibly included in the field; however, some items, such as the graphic log column, may be reserved for completion in the office. The purpose of the log is to clearly document the events and findings of the drilling activity. All pertinent data related to boring/drilling operations must be concisely recorded as objectively as possible. The subcontractor has the option to resubmit this form in a deliverable as a completely redrafted/typed form, or as a combination of information applied in the field and in the office. Regardless, the original field log should be retained in the permanent file. Any alterations or changes between the office copy and the original should be justified. To complete the boring or well logs:

- Fill out information on header of the log noting either boring or well number, if well is to be installed. Use the sampling site identification number.
- Note number under "Location".
- Note start and end date of boring or well installation under "Date", use MM/DD/YY format.
- Briefly describe wind direction, speed, and temperature under "Weather".
- The logging geologist should include his name under "Logged by", include three initials.
- The driller's name and drilling company should be included under "Drilled by", include three initials for the driller's name.
- "Drilling method" should contain information such as rotasonic drill diameter.
- "Sampling method" should be described as length of sampler and type, i.e., 2.5' split spoon.
- The sampling method should be described.
- "Gravel pack" should include the depth interval of gravel pack installation, sieve filter size, and type, i.e., 50'-39', 20-40 Colorado silica.
- "Seal" The seal should describe the depth interval of seal above the gravel pack and type.
- The seal should also describe the depth interval of grout slurry.

Under the header of casing, the casing description will require the following:

- "Type" Schedule 40 polyvinyl chloride (PVC), stainless steel etc.
- "Diameter" The information supplied here will be reported in inches (usually 4 inch).
- "Length" The length of casing or riser should include stick-up at the surface.

Under the heading of screen, the well screen will require the following information:

- "Type" Same as in type above.
- "Slot" The screen slot size. For silts and fine-grained sands, the slot size will be 0.01 inch.
- For sands medium to coarse grained, the slot size will be 0.02 inch.
- "Diameter" The diameter for well screens reported in inches (usually will be 4 inch).
- "Length" The length of the well screen in reported feet.
- "Hole Diameter" The diameter of hole cut by either a rotating bit or auger cutting head. Reported in inches.

- "Total Depth" The total depth drilled (in feet). If sampled deeper than depth drilled, this should be noted at the bottom of the log.
- "Location Map" A sketch of the boring location should be constructed in this corner.
- Topographical setting

Below the header are lithology/remarks and sample classifications. The following sample classifications should be described as follows:

- "Moisture Content" (Clays and Sands)
  - Dry
  - Damp
  - Moist (compactable)
  - Wet (not Compactable)
  - Saturated
- "Sorting" (Sands only)
  - Very well
  - Well
  - Moderately
  - Poorly
  - Very poorly
- "Density" or consistency (CONSS) (Sands and Clay) Density is described by the number of drops required by a 140 lb. hammer over 30 inches to drive a 2-inch outside diameter, 1 3/8 inch inside diameter, split-spoon 6 inches.
- Other descriptions may include:
  - NC (Noncemented)
  - PC (Poorly cemented)
- "Plasticity" Plasticity refers to the case in which cohesive soils are molded. The following describes the plasticity terms.
  - EXTREMELY HARD, resistant to pressure, not broken by hand
  - NONPLASTIC, not wire formable
  - SLIGHTLY PLASTIC, wire formable but soil remains easily deformed
  - PLASTIC, wire formable, moderate pressure required
  - VERY PLASTIC, wire formable, much pressure required
- "Sample Number" In this column, record the number order that the sample was taken.
- "TIP Reading" refers to "Total Ionizables Present". Record the headspace reading here and the type of instrument used, i.e., HNU, OVM, etc.
- "Sample Recovery" After obtaining a split-spoon sample or Shelby sample, measure the length of recovered sample to the nearest 0.01' and record level.
- "Penetration Resistance" The blow counts for every 6 inches of driving the sample are to be recorded under this heading.
- "Color"

## 6.0 REQUIRED FORMS

### 6.1 Drilling Log

### 6.2 Field Log Book

## **1.0 PURPOSE**

This Standard Operating Procedure (SOP) establishes guidelines and procedures for conducting field quality control (QC) sampling. Field QC sampling is required to assist in verifying the quality and integrity of samples collected during a given sampling event. Additional specific field QC sampling procedures and requirements will be provided in the project work plans.

## **2.0 REFERENCES**

2.1 EPA, September 1987, Compendium of Superfund Field Operations Methods, EPA 540/P-87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidance for Conducting Remedial Investigation and Feasibility Studies Under CERCLA, Interim Final OSWER Directive 9355.3-01.

## **3.0 RESPONSIBILITIES**

3.1 The Field Team Leader is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The Quality Assurance System Manager (QCSM) is responsible for periodic review of field generated documentation associated with this SOP. The QCSM is also responsible for implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to QC sampling requirements, issuing nonconformances, etc.) if problems occur.

3.3 The Field Team Lead(s) assigned to environmental and QC sampling activities are responsible for completing their tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from procedures to the Field Team Leader.

## **4.0 DEFINITIONS/MATERIALS**

### **4.1 Field QC Sample**

A field QC sample is a physical sample collected during or for a specific sampling event. The purpose of this sample is to evaluate the quality and integrity of original samples collected during the specific sampling event.

## **5.0 PROCEDURE**

This section contains the requirements for field QC sampling. Field QC sampling is required to provide data to verify the quality and integrity of environmental samples collected during a given sampling event.

The details within this SOP should be used in conjunction with project plans. These plans will generally provide the following information:

- Sample collection objectives
- Numbers, types and locations of environmental (non-QC) samples to be collected
- Numbers and types of supportive QC samples to be collected
- Any additional QC sampling requirements or procedures beyond those covered in this SOP, as necessary.

## 5.1 Quality Control Sampling Requirements

5.1.1 Field QC samples may consist of different media. Typical QC samples are as follows:

- Trip blank (TB)
- Equipment rinsate (ER)
- Field blank (FB)
- Field duplicate (FD)

5.1.1.1 Trip blanks are analyte-free water, shipped from and returned unopened to the laboratory in the same shipping containers for volatile organic analyses, and at times gasoline hydrocarbons analyses. The blanks are prepared at the laboratory using ASTM Type II DI Water, sent to the project location, carried with the sampling team(s) during sampling, and shipped to the laboratory for analysis with the environmental samples.

Trip blank samples are commonly collected and analyzed at a rate of one per sample cooler containing samples for volatile organic analyses or the gasoline fraction of petroleum hydrocarbons. The number or rate of trip blanks to be collected and the specific analyses to be conducted for the trip blanks will be provided in the project work plans.

5.1.1.2 Equipment rinsate samples are collected from the final rinse water during decontamination of groundwater, soil, or waste sampling equipment. This type of equipment includes bailers, split spoon samplers, soil sample sleeves, hand augering equipment, surface soil sampling equipment, purge and sample pumps, etc.

Rinsate samples are generally collected at a rate of one per day per sampling team during the sampling event. Equipment rinsates are usually collected from dedicated sampling equipment only upon installation. The number or rate of equipment rinsate samples to be collected for a particular project will be specifically developed and documented in the project work plans. The specific chemical analyses to be conducted for the rinsate samples will also be developed and documented in the project work plans.

5.1.1.3 Field blanks are prepared from the water which is used for decontamination. One sample from each sampling event and each water source or lot number is generally collected and analyzed for all parameters of interest for the project. Upon collection, a description of the water source for the field blank sample should be documented in the Sample Collection Log.

The number or rate of field blank samples to be collected for a particular project will be specifically developed and documented in the project work plans. The specific chemical analyses to be conducted for the field blank samples will also be developed and documented in the project work plans.

5.1.1.4 For soils, field duplicate samples are generally collected by co-located sampling (e.g., using successive sample tubes from the same split spoon sampling run) or by splitting samples. Field duplicate water samples are commonly collected by retaining consecutive samples from the sampling device (e.g., bailer or sample pump discharge line). Field duplicate water samples may also be generated by splitting a collected volume; however, this practice may lead to a loss in volatile organic compounds and is not common practice for volatile analyses.

Field duplicate samples are commonly collected at a rate of 10 percent per media sampled. However, the number or rate of field duplicate samples to be collected for a particular project will be specifically developed and documented in the project work plans. The specific chemical analyses to be conducted for the field duplicates will also be developed and documented in the project work plans.

5.1.2 The type and number of QC samples collected for a particular project is based on specifications provided in project specific documents, i.e., the project work plans. Field QC samples are to be collected at appropriate times during a sampling event.

5.1.3 All field QC samples will be collected in proper containers with appropriate preservation per the project work plans.

5.1.4 The collection of field QC samples consisting of various media (e.g., soil, groundwater, etc.) will follow procedures in sample collection SOPs for the respective media and any other applicable procedures in the project work plans. For example, the collection of a groundwater field duplicate QC sample will follow procedures specified in the groundwater sampling SOP (SOP-17). Equipment rinse samples are collected directly while rinsing the sampling equipment following appropriate procedures in SOP-17 and the project work plans. Field blank samples are collected by pouring decontamination water directly into sample containers following appropriate protocol in SOP-17 and the project work plans.

5.1.5 Field QC samples will be labeled and numbered as described in SOP-04 and 05, respectively and the project work plans.

5.1.6 The field QC samples will also be maintained under custody per SOP-01 and be appropriately stored, handled and shipped per SOP-03 and 06.

## **6.0 REQUIRED FORMS**

6.1 Drilling Log

6.2 Chain of Custody Form

6.3 Field Log Book

## **1.0 PURPOSE**

Wastes covered by this policy statement include wastes generated from the investigation. Analysis of waste performed using this SOP are to determine the proper hazardous waste characterization and insure the waste is disposed of consistent with all applicable Solid and Hazardous Waste Regulations. Prior to disposal, the wastes will be collected, transferred and stored in accordance with all applicable regulations.

## **2.0 REFERENCES**

2.1 EPA, 1991. Management of Investigation – Derived Wastes During Site Inspection. OERR Directive 9345.3-02. May.

2.2 EPA, 1992. Guide to Management of Investigation Derived Wastes. OERR Directive 9345.3.03FS. January.

## **3.0 RESPONSIBILITIES**

Investigation Contractor - This is any contractor completing the environmental investigation. Examples of such investigations would include RCRA Facility Investigations, Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) assessments and investigations, and groundwater monitoring programs under the direction of either EPA or the State of Arizona.

The contractor(s) are tasked with the collection, on-site storage, transportation and disposal of solid and hazardous waste generated from the site investigation and remediation work in accordance with all applicable regulations. This includes the proper containerization and labeling of wastes; safe movement of waste containers; characterization of the waste; temporary storage of the waste; and the preparation of the hazardous waste manifests. Unless the waste has been previously characterized, all containers will be sampled within 10 days of being filled. The sample will be analyzed, as discussed in the paragraphs below, and characterized for proper disposal within 30 days of being sampled. The waste will then be disposed of accordingly within 30 days of completing the characterization. Once an analysis has been completed, the waste will be characterized and disposed of based on the results of that analysis.

All off-site waste disposal (both non-hazardous and hazardous, if required) must be coordinated with the Client RCRA Manager for tracking purposes.

## **4.0 PROCEDURES**

### **4.1 Waste Minimization Procedures**

To the extent that it is practical, the Investigation Contractor will follow waste minimization procedures during environmental investigations. Guidelines for waste minimization are:

- Minimize materials which are introduced into any exclusion zone in an investigation area.
- Combine similar wastes throughout an investigation area in a single container wherever possible.

- Combine decontamination water from multiple sites in one container.
- Use a container of the appropriate size (e.g., use a 5-gallon drum for a small amounts of waste unless a 55-gallon drum is needed to hold all the waste).
- Decontaminate and reuse material and equipment whenever practical. Minimize the volume of decontamination water generated.
- With solid environmental media and materials, ensure that waste is tightly packed to minimize the number of containers.
- Use less hazardous substances whenever possible.

#### 4.2 Waste Containerization and Labeling

Investigation or remediation derived waste, which are known or suspected to be hazardous waste, will be placed in appropriate DOT containers, supplied by the contractor, and labeled as hazardous waste.

##### 4.2.1 Labels

4.2.2 Labels for shipping waste on public roads will be supplied by the contractor. Upon filling of a container, the start accumulation date will be annotated on the label and the container will be transferred off-site within 3 days.

#### 4.3 Waste Management and Sampling Procedures

Wastes are either managed in open top, or closed top drums, gondolas, or in some instances discharged to a bulk tanker for transport to a Treatment Storage and Disposal Facility (TSDF). The sampling method selected for a given waste stream is based on the physical properties the waste exhibits. Liquids will be sampled with a coliwasa or glass tube; dry powder, sludges, and moist granules will be sampled with a trier; and packed powder will be sampled with an auger.

Each sample will be taken using a sampling tool that will insure the most representative sample. When more than one container is generated per waste stream, the sample to be analyzed will be a composite sample comprised of equal amounts taken from all the containers filled with that waste stream. For example, the drill cuttings from a single well would be composited into one sample. However, if information pertaining to the waste stream indicates the contamination may vary significantly, then composites would only be utilized for those portions of the waste stream with similar characteristics or each container would be sampled.

#### 4.4. Waste Characterization

Unless the waste has been previously characterized, all containers will be sampled within 10 days of being filled. The sample will be analyzed, as discussed in the paragraphs below, and characterized for proper disposal within 30 days of being sampled. The waste will then be disposed of accordingly within 30 days of completing the characterization.

#### 4.5 Parameter Test Methods

The type of analysis of each waste will depend upon the operations previously conducted at the site and information gained from previous investigative or remedial work performed. The parameters of analysis that normally will be considered include the characteristics of Ignitability, Corrosivity, Reactivity, Toxicity Characteristic Leaching Procedure (TCLP) Metals, TCLP

Pesticides/Herbicides and TCLP Organics. Parameters for F and K listed hazardous wastes will only be analyzed for if information specific to the site indicates their possible presence. Parameters will be eliminated when previously gathered information for a site or the physical state of the waste generated would so justify. For example, if the waste were a solid, then the parameters of ignitability and corrosivity would be eliminated.

## **5.0 REQUIRED FORMS**

5.1 Investigation Derived Waste Log

5.2 Field Log Book

Refer to attached Instruction Manual for the X-Ray Fluorescence Spectrometers, in particular Chapter 4 for Operating Instructions.

# **INSTRUCTION MANUAL**

## **INNOV-X SYSTEMS ALPHA SERIES™ X-RAY FLUORESCENCE SPECTROMETERS**

**August 2005  
Version 2.1**

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# Chapter 1 Introduction

## 1.0 INSPECTING YOUR INNOV-X ANALYZER

### Upon receipt:

1. Locate and remove the shipping papers and documentation from under the lid's foam padding.
2. Remove the Innov-X Analyzer and all of the components from the protective carrying case and identify each on the enclosed shipping list.
3. Connect the battery charger to an 110V-240V AC power source. Place one Li-ion battery on the charger and charge it for at least 2 hours. Charge the second battery.
4. Charge the HP iPAQ using the attached AC adaptor for at least ½ hour.
5. Read and review the "Quick Start" section of the User's Manual. Innov-X recommends that you read the entire manual.
6. Install the fully charged battery into the analyzer.
7. Press the ON/OFF button on the back of the analyzer and the power button on the iPAQ.
8. Select Innov-X from the start menu located in the upper left hand corner of iPAQ screen.
9. Select the desired analysis mode (i.e., Analytical, FastID, Pass/Fail or Soil). The instrument will undergo a one minute hardware initialization period.
10. Standardize the instrument with the 316 Stainless Steel mask. Standardize the instrument every 4 hours or as directed by the display.
11. Release the software trigger lock and analyze a sample of known composition, in order to verify the correct operation of the analyzer.
12. Analyze samples of unknown composition.

## 1.1 COMPONENTS INCLUDED WITH THE ANALYZER

Shown here are the various items which are included with the Innov-X portable XRF analyzer. Unless otherwise noted, all items are standard accessories.



Analyzer, with iPAQ attached.



Two, Li-ion batteries (one shown).



Battery charger and an AC adaptor. Battery shown mounted in charging system.



Standardization cap and weld mask (optional)

The standard standardization cap has no weld slit.



iPAQ cradle and AC adaptor. The cradle is used to connect the iPAQ to a PC for downloading data and reports.



Testing stand. This is the benchtop docking station for the analyzer. It is an optional accessory

## 1.2 QUICK START INSTRUCTIONS

The following section provides a quick overview to using the Innov-X portable XRF analyzer. This is intended to provide the basic startup and operational instruction needed to perform simple analyses. It is highly recommended that the user read the sections on Radiation Safety (Chapter 3) and the detailed description on operation (Chapter 4). The following Quick Start information is also provided as a separate, bound, laminated publication for quick reference.

1. Place a battery in the analyzer.
2. Power on the Analyzer (On/Off switch located on back of analyzer)
3. Power on the iPAQ (Button located in upper right hand corner of iPAQ)
4. Select Innov-X from the start menu located in the upper left hand corner of iPAQ screen.
5. Read the radiation safety notice and acknowledge that you are a certified user by pressing Start.
6. Select Desired Mode.
7. The analyzer will undergo a 60 second hardware initialization.
8. Place a standardization clip on the nose of the analyzer. Tap the button on the screen to standardize. (*Manual section 4.4 Standardization*)
9. When standardization is complete, remove the standardization clip.

10. Release the software trigger lock by tapping the locked icon on the iPAQ screen and tapping yes in response to the software prompt.
11. Test standard to verify instrument performance.
12. Results will display on screen. Subsequent tests may be started from either the Results or Analysis screens.

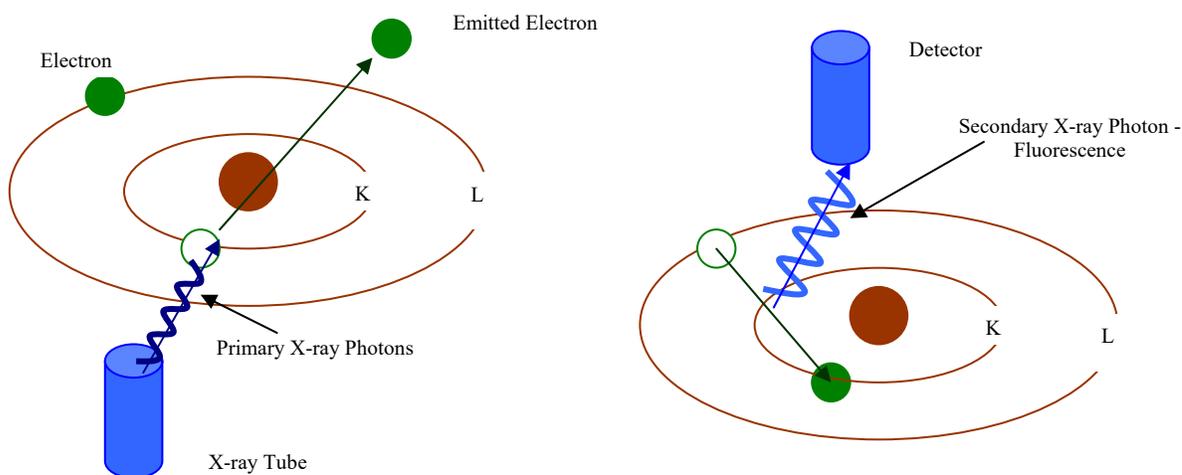
## 1.3 INTRODUCTION TO XRF: X-RAY FLUORESCENCE SPECTROMETRY OVERVIEW

### Basic Theory

Although most commonly known for diagnostic use in the medical field, the use of x-rays forms the basis of many powerful analytical measurement techniques, including X-ray Fluorescence (XRF) Spectrometry.

XRF Spectrometry is used to identify elements in a substance and quantify the amount of those elements present. An element is identified by its characteristic X-ray emission wavelength ( $\lambda$ ) or energy (E). The amount of an element present is quantified by measuring the intensity of its characteristic line. XRF Spectrometry ultimately determines the elemental composition of a material.

All atoms have a fixed number of electrons (negatively charged particles) arranged in orbitals around the nucleus. The number of electrons in a given atom is equal to the number of protons (positively charged particles) in the nucleus; and, the number of protons is indicated by the Atomic Number in the Periodic Table of Elements. Each Atomic Number is assigned an elemental name, such as Iron (Fe), with Atomic Number 26. Energy Dispersive (ED) XRF and Wavelength Dispersive (WD) XRF Spectrometry typically utilize activity in the first three electron orbitals, the K, L, and M lines, where K is closest to the nucleus. Each electron orbital corresponds to a specific and different energy level for a given element.



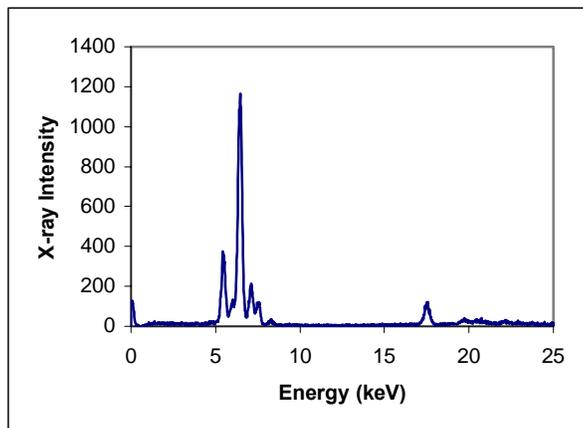
In XRF Spectrometry, high-energy primary X-ray photons are emitted from a source (X-ray tube) and strike the sample. The primary photons from the X-ray tube have enough energy to knock electrons out of the innermost, K or L, orbitals. When this occurs, the atoms become ions, which are unstable. Electrons seek stability; therefore, an electron from an outer orbital, L or M, will move into the newly vacant space at the inner orbital. As the electron from the outer orbital moves into the inner orbital space, it emits an energy known as a secondary X-ray photon. This phenomenon is called fluorescence. The secondary X-ray produced is characteristic of a specific element. The energy (E) of the emitted fluorescent X-ray photon is determined by the difference in energies between the initial and final orbitals of the individual transitions.

This is described by the formula

$$E=hc/\lambda$$

where h is Planck's constant; c is the velocity of light; and  $\lambda$  is the characteristic wavelength of the photon.

Wavelengths are inversely proportional to the energies; they are characteristic for each element. For example the  $K\alpha$  energy for Iron (Fe) is about 6.4keV. The number of element-specific characteristic X-rays produced in a sample over a given period of time, or the intensity, can be measured to determine the quantity of a given element in a sample. Typical spectra for EDXRF Spectrometry appear as a plot of Energy (E) versus the Intensity (I).



## History

Wilhelm Roentgen discovered X-rays in 1895. Methods for identifying and quantifying elements using XRF were first published by Henry Moseley in 1913. Much research and development of XRF continued after Moseley's pioneering work, especially during WWII when rapid developments in the aircraft, automotive, steel and other metals industries heightened the need to identify alloys quickly and reliably. However, the first commercial XRF Spectrometers weren't available until the early 1950's. Those systems were based on WDXRF technology and measured the characteristic wavelength of an element, one element at a time. Although the use of these systems was critical for elemental analyses, they were large, expensive, and required highly skilled operators to use and maintain them.

In the late 1960's, EDXRF technology, which measures the characteristic energy of an element, began to rival the use of WDXRF due to the development of Si (Li) solid state detectors, which offered better energy resolution of the signal. EDXRF systems offered the potential of collecting and displaying information on all of the elements in a sample at the same time, as opposed to one at a time with typical WDXRF systems. Many of the early EDXRF systems used radioisotopes for excitation instead of X-ray tubes, which could require changing sources to determine all the elements of interest. Some of those early EDXRF systems did not easily resolve multiple elements in a single analytical run.

As can be imagined, the equipment and applications of XRF Spectrometers have developed tremendously since the 1960's. Advancements in technology, electronics, computers, software and the use and modification of them for XRF Spectrometers by instrument manufacturers, research scientists & engineers, and industrial users alike have led to the current state of the art in XRF Spectrometers. Now a mature technology, XRF Spectrometry is routinely used for R&D, QC and analytical services in support of production.

## Elemental Analysis

XRF Spectrometry is the choice of many analysts for elemental analysis when compared to the other techniques available. Wet chemistry instrument techniques for elemental analysis require destructive and time-consuming specimen preparation, often using concentrated acids or other hazardous materials. Not only is the sample destroyed, waste streams are generated during the analytical process that need to be disposed of, many of which are hazardous. These wet chemistry elemental analysis techniques often take twenty minutes to several hours for specimen preparation and analysis time. All of these factors lead to a relatively high cost per sample. However, if PPB and lower elemental concentrations are the primary measurement need, wet chemistry instrument elemental analysis techniques are necessary.

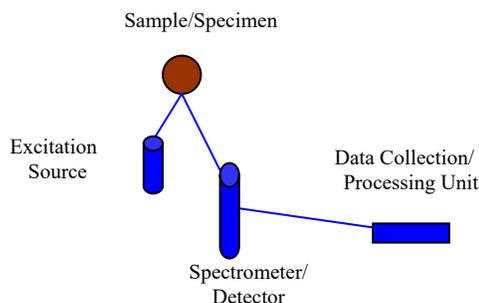
XRF Spectrometry easily and quickly identifies and quantifies elements over a wide dynamic concentration range, from PPM levels up to virtually 100% by weight. XRF Spectrometry does not destroy the sample and requires little, if any, specimen preparation. It has a very fast overall sample turnaround time. These factors lead to a significant reduction in the per sample analytical cost when compared to other elemental analysis techniques.

All elemental analysis techniques experience interferences, both chemical and physical in nature, and must be corrected or compensated for in order to achieve adequate analytical results. Most wet chemistry instrument techniques for elemental analysis suffer from interferences that are corrected for by both extensive and complex specimen preparation techniques, instrumentation advancements, and by mathematical corrections in the system's software. In XRF Spectrometry, the primary interference is from other specific elements in a substance that can influence (matrix effects) the analysis of the element(s) of interest. However, these interferences are well known and documented; and, instrumentation advancements and mathematical corrections in the system's software easily and quickly correct for them. In certain cases, the geometry of the sample can effect XRF analysis, but this is easily compensated for by grinding or polishing the sample, or by pressing a pellet or making glass beads.

Quantitative analysis for XRF Spectrometry is typically performed using Empirical Methods (calibration curves using standards similar in property to the unknown) or Fundamental Parameters (FP). FP is frequently preferred because it allows elemental analysis to be performed with no standards or calibration curves. This enables the analyst to use the system immediately, without having to spend additional time setting up individual calibration curves for the various elements and materials of interest. The capabilities of modern computers allow the use of this no-standard mathematical analysis, FP, accompanied by stored libraries of known materials, to determine not only the elemental composition of an unknown material quickly and easily, but even to identify the unknown material itself.

## EDXRF Spectrometers

EDXRF Spectrometer systems are mechanically very simple; essentially there are no moving parts. An EDXRF system typically has three major components: an excitation source, a spectrometer/detector, and a data collection/processing unit. The ease of use, rapid analysis time, lower initial purchase price and substantially lower long-term maintenance costs of EDXRF Spectrometers have led to having more systems in use today worldwide than WDXRF Spectrometer systems.



EDXRF has been found most useful for scrap alloy sorting, forensic science, environmental analysis, archaeometry and a myriad of other elemental field-oriented analyses.

## **Handheld EDXRF Spectrometers for Field Analyses**

It is clear that a future trend for elemental analysis is in rapid site investigation using techniques that are fast, inexpensive, reliable, and long-term cost effective. There is a need for immediate decisions to be made during the delivery of materials, industrial processing, and in the field for positive materials identification or environmental site assessment and remediation. It is also clear that EDXRF Spectrometry is the most suitable elemental analysis technique available for field analysis due to its simplicity, speed, precision, accuracy, reliability, and overall cost effectiveness.

Recent technological developments in cell phones, pocket PC's and other portable consumer electronics have led to the advancement of many high-performance, miniature components. X-ray equipment manufacturers began to take advantage of these developments in the late 1990's and developed Handheld EDXRF systems. An obvious advantage of Handheld EDXRF systems is that the analyzer is taken to the sample as opposed to bringing the sample to the analyzer and configuring it to fit in an analysis chamber. In addition to the per sample analytical cost savings, a key factor in using non-destructive EDXRF analysis, especially in the field, is the overall project cost savings due to improved and more timely decision making. The use of EDXRF for immediate positive materials identification or to guide an environmental site characterization will generally reduce the overall time required in the field due to the quick turnaround for the sample analysis; this invariably reduces the overall costs of analytical field work.

Of course, Handheld EDXRF technology has continued to evolve in concert with portable consumer electronic developments. Just like the early Benchtop EDXRF systems, early Handheld EDXRF systems used radioisotopes for excitation. There are several practical problems with the use of radioactive isotopes for handheld systems. The source decays and loses its testing speed over time. In addition to the loss in analytical capabilities, the sources have to be replaced incurring a cost. The use of radioactive isotopes also requires licensing (state-to-state in the US) and a radioactive materials control program; they are difficult to ship and transport, as they require hazardous materials declarations and/or permits. Consequently, the newest and most exciting development in Handheld EDXRF technology is the use of battery operated, miniature X-ray tubes, which was pioneered by the staff at Innov-X Systems.

## **Innov-X Systems Handheld EDXRF Spectrometers**

Innov-X Systems specializes in Handheld EDXRF technology with the most advanced miniature components available for X-ray Tube sources, detectors, and PC 's. Innov-X Systems Handheld EDXRF Spectrometers are ideally suited for field analysis of alloys, lead-based paint, environmental soils, filters, dust wipes, forensics, archaeometry, and a variety of other elemental analyses in the field or around the plant. Innov-X Systems EDXRF Spectrometers are affordable, easy to use, reliable, and overall cost effective. The Innov-X Systems Handheld EDXRF units incorporate state-of the art components including a battery operated miniature X-ray tube, a high-resolution silicon pin detector, high speed data acquisition circuitry, and a Compaq IPAQ Pocket PC<sup>®</sup> handheld computer for calculations, results and operator interface.

Innov-X Systems EDXRF Spectrometers offer the following invaluable features:

- Portable
- Battery operated, rechargeable
- X-ray Tube-based (Ag or W anode, 10-40kV, 10-100uA)
- Si PiN diode detector.
- Integrated pocket PC
- Pistol-shaped design for difficult testing locations and welds
- Auto-compensation for irregular or small samples
- Fundamental Parameters for no-standard analyses
- Stored Grade Libraries for rapid Grade ID's
- Stored Fingerprint Libraries for rapid material ID's

- Docking station available for use as standard benchtop unit
- Results shown after a few seconds of testing time.

For more information on how to utilize your Innov-X Systems Handheld EDXRF Spectrometer optimally, please review this Instruction Manual or contact us directly.

# Chapter 2. Usage and Assembly of Accessories

## 2.0 ACCESSORIES

This chapter describes the various accessories that are provided with an Innov-X XRF analysis system. Included are:

- Batteries
- Battery Charger
- iPAQ cradle and charger
- Testing Stand Assembly (not standard with all units)
- Standardization Clip or Standardization Clip/Welding mask.

## 2.1 ANALYZER BATTERY

The Innov-X Systems XRF Analyzer is powered by a replaceable, rechargeable Lithium ion battery. In addition, the iPAQ has its own internal battery.

### Innov-X Systems Main Battery

The Innov-X Analyzer uses a rechargeable Lithium Ion Smart Battery. A picture of the battery is shown in Fig. 2.1. Two batteries are included with each analyzer. The batteries are charged an external battery charger. Batteries typically function for 4 to 8 hours, depending on usage patterns. Heavier duty cycles deplete the battery more quickly. Therefore, users who do longer and more frequent tests will need to replace their batteries more often than users who take shorter or fewer tests.



Figure 2.1. Li-ion Battery for analyzer

Replacement batteries can be purchased directly by calling Innov-X Systems at 781-938-5005. (P/N A003)

### Battery power indicators:

There are two ways of determining the charge remaining on a battery: the LED indicator on the battery and the battery status icon on the analyzer screen. The battery icon, when tapped, will indicate the percent charge remaining on a battery inside the analyzer. Additionally, the battery icon will change from green to yellow when the battery gets low, indicating it has about 15 minutes left of charge.

To use the battery LED, push the button below the indicator. The lighting will indicate the % of charge. If possible, try to use batteries with at least 50% of their full charge, according to the indicator.

## 2.2 CHANGING A BATTERY

To change a battery, perform the following steps:

1. Hold the instrument by the handle, upside down, so the bottom of the instrument base is pointing upward. Please refer to Fig. 2.2.
2. Hold the instrument so that the nose is pointing away from the operator.
3. Open the battery door on the bottom of the handle. The batteries have a small tab attached for ease of removal.

4. Pull out the existing battery, and replace with a new battery.
5. Insert the charged battery into the analyzer such that the connectors on the top of the battery are facing to the right. Note that the battery slot is keyed so that the battery can only be inserted one way.



Figure 2.2a. Instrument handle. Pull the rubber latch and lift door. Reach into opening and remove battery.



Figure 2.2b Insert new battery into opening.

## 2.3 BATTERY CHARGER

The battery charger is shown in Fig. 2.3. It takes about 2 hours to completely charge a battery. The status of the charger is shown by two lights on the power adaptor. Table 2.1 lists the information conveyed by the lights.



Figure 2.3. Battery charger.

Left Light	Right light	Status
On	Off	Battery is charging
On	On	Battery is 80% charged
Off	On	Battery is completely charged
Blink	Blink	Error. Remove battery and replace on charger. If error persists, call Innov-X Systems Technical support.
Off	Off	No battery is on charger

Table 2.1 Battery charger status lights

## 2.4 HP IPAQ POCKET PC BATTERY

The iPAQ has an internal rechargeable battery, which can be recharged by using the power adaptor that is included with the unit. This adaptor can be connected either to the iPAQ itself, or to the cradle. If it is connected to the cradle, and plugged in, the iPAQ will recharge whenever it is placed in the cradle. In addition, the iPAQ Battery will recharge whenever the iPAQ is mounted in an Innov-X analyzer which is powered, but not actively taking a test. The amber light on the top of the iPAQ will blink whenever the battery is charging. It will remain solid when the battery is completely charged.

Since the iPAQ will be recharged whenever the Innov-X Systems Analyzer is in use, it may never be necessary to use the iPAQ power adaptor. However, care should be taken when the analyzer is not used for a period of several days, as the iPAQ uses some power even when it is powered off. It is therefore possible to completely discharge the battery simply by not using the iPAQ for several days, or by using it for several hours without recharging it.

If you do not use your Innov-X Analyzer on a daily basis, or if you will have a down period of more than several days, it is recommended that you remove the iPAQ from the Analyzer when it is not in use and plug in the iPAQ to a power outlet to recharge it. This will ensure that your iPAQ is always charged and ready for use. You should also always plug in the power cord whenever the iPAQ is removed from the analyzer for data transfer.

If you do allow the iPAQ battery to discharge significantly, either by allowing it to sit too long unused, or by using it for a period of time without it being connected to a power source, it may not be possible to operate your analyzer. If this happens, the Innov-X software will provide an error message indicating that the iPAQ battery is too low. Recharge the iPAQ for at least a half an hour before attempting another measurement.

If the iPAQ battery is completely discharged, it will not be possible to turn on the iPAQ until it is recharged. A complete power failure will erase anything that is stored in the Main Memory of the iPAQ. All Innov-X program and data files are stored on the storage card, rather than in Main Memory, so you will not lose any data or have to reinstall the Innov-X software.

1. If the battery on the iPAQ is completely discharged, charge it for at least one half hour.
2. You will be required to follow the prompts on the iPAQ screen before you can use the iPAQ. This procedure involves realigning the screen by tapping in several spots, and going through a quick tutorial.
3. The iPAQ will reinitialize the Innov-X Systems software. A message will appear indicating that this is going to happen. You must tap ok to initialize.
4. The software will open automatically; a message will appear indicating that several registries have been restored. Tap ok to dismiss this message.
5. Set the clock to the current time. **Note, this is very important**, as your data is indexed by date. If the date in the iPAQ is incorrect, you may not be able to locate your results. The instrument will not allow you to take a reading until the date has been changed.
  - a. From the Start Menu, tap Settings.
  - b. Select the System tab, and tap clock.
  - c. Set the proper date. Further details about this procedure can be found in the HP iPAQ user's manual.

## 2.5 REMOVING THE IPAQ FROM THE ANALYZER

It is very important to properly remove the iPAQ Pocket PC from the analyzer to avoid damaging the connector on the back of the iPAQ.

In order to remove the iPAQ, push the iPAQ retainer shown in Fig. 2.4 towards the front of the analyzer. Holding the retainer forward, grab the iPAQ from the sides, slide the iPAQ forward until it is clear of its

connector, then tilt the front end up enough so it clears the front holder allowing the iPAQ to be lifted out of the instrument.

**Note:** Never grab the iPAQ and twist it side-to-side to remove it from the analyzer. Always move the iPAQ retainer forward as instructed above, slide the iPAQ forward and remove from the analyzer.



Figure 2.4. Removing the iPAQ from the analyzer.

## 2.6 STANDARDIZATION CAP and/or WELD TESTING MASK

All analyzers are supplied with either a standardization cap or a combination standardization cap welding mask. The standardization mask is the standard accessory. Welding masks can be purchased as an additional accessory, or in lieu of the standardization mask.

### Standardization Cap

The cap clips on the front end of the analyzer and is used to standardize the system as described in Chapter 4. To attach the cap, snap it onto the nose of the analyzer over the Kapton window.

### Combination Standardization Cap/Welding Mask

The standardization/welding mask is shown in Fig. 2.5. The cap clips onto the front end of the analyzer and is used to standardize the system as described in Chapter 4. To attach the cap, snap it onto the nose of the analyzer over the Kapton window. Be sure that when attaching the cap, that the solid end (as opposed to the end with the 1/4" wide slit) is covering the window. To remove the mask, slide it off to either side.

The opposite end of the standardization cap serves as a welding mask. This mask is used to shield the base metal from analysis, when analyzing a weld. It is important to use this mask since failure to do so will produce an alloy chemistry that is a mixture of the base metal and the actual weld. For best results:

- a. Use the welding mask only for welds that are larger than the opening in the mask;
- b. Make solid contact between the surface of the mask and the material to test;
- c. Use the mask only in the Analytical Mode – not with the standard Fast ID library;
- d. Consider using longer test periods to compensate for the smaller testing area – especially with more difficult separations.

If it is desirable to use the welding mask in FastID mode, a user can create a special “Welding Mask Library.” Teach all relevant alloys with the welding mask in position. Make sure these fingerprints are

saved in library that contains ONLY fingerprints taught with a welding mask. When measuring a weld, make sure the “Weld” library is the only one selected. By creating a special finger print library using the welding mask, a user can get good results in the Fast ID Mode as well.



Figure 2.5 Standardization cap and welding mask. (Optional accessory)  
The standard standardization cap does not have the welding slit.

## 2.7 TESTING STAND (optional accessory)

The testing stand is designed as a docking station for the handheld analyzer. It can be used as a bench-top system, or to test small samples. A list of components and an assembled stand is shown in Figure 2.6:



Figure 2.6. Assembled Testing Stand

### Components of the testing stand:

1. Three (3) short legs
2. Three (3) long legs
3. Lower Stand
4. Upper Stand
5. Four (4) knobs for top plate
6. Test stand cradle
7. Clip for cradle.
8. Adaptor cable (connects serial connector on iPAQ cradle to auxiliary port on analyzer)

### Assembly of Testing Stand

1. Insert the three Short Legs through the holes in the Lower Stand by inserting the threaded screw through the holes. This will balance the Lower Stand on the table top. (Fig. 2.7).



Figure 2.7. Mounting Lower Stand onto Short Legs.

2. Mount the three Long Legs onto the Lower Stand by inserting the threaded screws from the Short Legs into the holes on the Long Legs and turning until snug. Remove iPAQ from analyzer by following the instructions in Figure 2.4. Place the analyzer into the gap in the Lower Stand as shown. (Fig. 2.8).



Figure 2.8. Mounting Long Legs onto Lower Stand and inserting analyzer.

3. Mount the Upper Stand onto the Long Legs. The Upper Stand has holes for the screws at the end of each of the Long Legs. The Upper Stand will also fit snugly over the front end of the analyzer. Be sure that the Upper Stand is mounted so that all three screws are inserted through the holes, and the front end of the analyzer is flush with the top surface of the upper stand. (Fig. 2.9).



Figure 2.9. Mounting Upper Stand onto Testing Stand.

5. Put three knobs to secure testing stand onto analyzer. The iPAQ clip can be secured with any of the knobs. This clip grabs the base of the iPAQ cradle to hold the iPAQ securely in place.

6. Place the iPAQ in the cradle and connect it to the Auxiliary Port on the analyzer using the serial cable adaptor.



Figure 2.10. Connecting iPAQ to Auxiliary Port on analyzer.

# Chapter 3 Safety Information

## 3.0 IMPORTANT SAFETY INFORMATION

**THE XRF SHOULD NOT BE POINTED AT ANYONE OR ANY BODY PART, ENERGIZED OR DE-ENERGIZED!** The safe and proper operation of the Innov-X XRF instruments is the highest priority. These instruments produce ionizing radiation and should **ONLY** be operated by individuals, who have been trained by Innov-X Systems, Inc. and received a manufacturer's training certificate. Innov-X recommends that operators and companies implement a written Radiation Safety Program, with safety components specific to the site and application of use of the instrument. The Radiation Safety Program should be reviewed annually and revised appropriately by a competent individual.

Innov-X analyzers must be used by trained operators, according to the instructions presented in this manual. Improper usage may circumvent safety protections and could potentially cause harm to the user. Pay attention to all warning labels and messages.

### **Important Notice for all Canadian Users:**

Canadian Federal Regulations (Radiation Emitting Devices Act) require that all Canadian users must be certified according to NRC Standard CAN/CGSB-48.97/2-2000 in order to use this device.

For this certification contact: Natural Resources Canada, Manager Nondestructive Testing Certification, CANMET, 568 Booth St., Ottawa, ON, K1A 0G1; Tele: (613) 943-0583; Fax(613) 943-8297.

Users are advised to contact their appropriate federal/provincial./territorial radiation protection agency for applicable rules of operation.

The Innov-X analyzer is a very safe instrument when used according to manufacturer's recommended safety procedures as detailed in this chapter.

Radiation levels during testing are  $< 0.1$  mR/hr on all surfaces of the analyzer except at or near the exit port for the radiation. This means that if an operator follows standard operating procedures, they will not obtain any detectable radiation dose above naturally occurring background radiation, on their hand while holding the analyzer, or on any area of their body.

This chapter details specifics of the radiation levels. It covers both standard (safe) and un-safe methods of operation, it provides radiation emission information, and also provides dose estimates for unsafe operations.

## 3.1 GENERAL SAFETY PRECAUTIONS AND INFORMATION:

Retain and follow all product safety and operating instructions. Observe all warnings on the product and in the operating instructions. To reduce the risk of bodily injury, electric shock, fire and damage to the equipment, observe the following precautions:

Heed service markings. Except as explained in this documentation, do not service any Innov-X product yourself. Opening or removing covers may expose you to electric shock. Service needed on components inside these compartments should be done only by Innov-X Systems, INC.

Damage requiring service:

- The power cord, plug or battery contacts for the battery charger are damaged.

- Liquid has been spilled or an object has fallen onto the instrument.
- The instrument has been exposed to rain or water.
- The instrument has been dropped or damaged.
- There are noticeable signs of overheating.
- The instrument does not operate normally when you follow operating instructions.

Safety Precautions:

Use the correct external power source: Ensure that the voltage is appropriate (100V-240 V/ 50-60 Hz) for charging the battery packs. Do not overload an electrical outlet, power strip, or convenience receptacle. The overall load should not exceed 80% of the branch circuit rating.

Use cables and power cords properly:

Plug the battery charger into a grounded electrical outlet that is easily accessible at all times. Do not pull on cords and cables. When unplugging the cord from the electrical outlet, grasp and pull the cord by the plug.

Handle battery packs properly; do not: disassemble, crush, puncture, short external contacts, dispose of in fire or water, or expose a battery pack to temperatures higher than 60 °C (140 °F). Do not attempt to open or service a battery pack.

**WARNING:** Danger of explosion if battery is incorrectly substituted. Replace only with Innov-X specified batteries. Used batteries may be returned to Innov-X Systems for disposal.

### 3.2 INNOV-X SYSTEMS – RECOMMENDED RADIATION SAFETY TRAINING COMPONENTS

Individual Companies and States have specific regulations and guidelines for the use of X-ray tube generated ionizing radiation. The purpose of the recommendations below is to provide generic guidance for an ALARA - best practice - approach to radiation safety. These recommendations do not replace the requirement to understand and comply with the specific policies of any state or organization.

1. **Proper Usage.** Never point the instrument at another person. Never point the instrument into the air and perform a test. Never hold a sample in your hand and test that part of the sample.
2. **Establish Controlled Areas.** The location of storage and use should be of restricted access to limit potential exposure to ionizing radiation. In use, the target should not be hand held and the area at least three paces beyond the target should be unoccupied.
3. **Specific Controls.** The instrument should be stored, in a locked case, or locked cabinets when not in use. When in use, it must remain in the direct control of a factory trained, certified operator.
4. **Time - Distance - Shielding Policies.** Operators should minimize the time around the energized instrument, maximize the distance from the instrument window, and shoot into high density materials whenever possible. Under no circumstances should the operator point the instrument at themselves or others.
5. **Prevent Exposure to Ionizing Radiation.** - All reasonable measures, including labeling, operator training and certification, and the concepts of time, distance, & shielding, should be implemented to limit radiation exposure to *as low as reasonably achievable* (ALARA).
6. **Personal Monitoring.** Radiation control regulations may require implementation of a radiation monitoring program, where each instrument operator wears a film badge or TLD detector for an initial period of 1 year to establish a baseline exposure record. Continuing radiation monitoring after this

period is recommended, but may be discontinued if accepted by radiation control regulators. Please refer to Sect. 3.10 for a list of providers of film badges.

### 3.3 INNOV-X SAFETY FEATURES

The Innov-X analyzer is very safe when used correctly, however the analyzer does emit radiation through the analyzer window, and all precautions must be taken to reduce exposure to this radiation. In order to minimize the possibility of accidental exposure, the following safety features are standard in all Innov-X analyzers.

1. “Deadman” trigger. The trigger must be held for the duration of the test. This requires that the user consciously depress the trigger whenever x-rays are emitted, and ensures that the analyzer is attended at all times while x-rays are emitted.

Upon completion of safety training, an INNOV-X certified trainer may deactivate this feature upon request. The deactivation of the trigger is recommended only if long tests are required (such as for soil mode) and if the unit is used primarily by only 1 or 2 users who utilize it frequently, in a very controlled environment. In situations where multiple users are sharing the unit, it is recommended that the deadman trigger remain active.

**Note: Canadian Regulations require that the deadman trigger be used at all times. This feature will not be disabled for usage in Canada.**

2. Software Trigger lock. Before using the trigger, the user must tap on a lock icon located in the lower right hand corner of the iPAQ screen. The user must then confirm that they wish to unlock the trigger. If the instrument is used continuously, the software trigger lock will remain off. If five minutes elapse between tests, the trigger will lock automatically.
3. Software Proximity sensor. The software requires that a sample be present in front of the analyzing window. This prevents the accidental exposure of bystanders to an open beam. If the analyzer detects that a sample is not present, it will abort the test and shut off x-rays two seconds after the test is started.

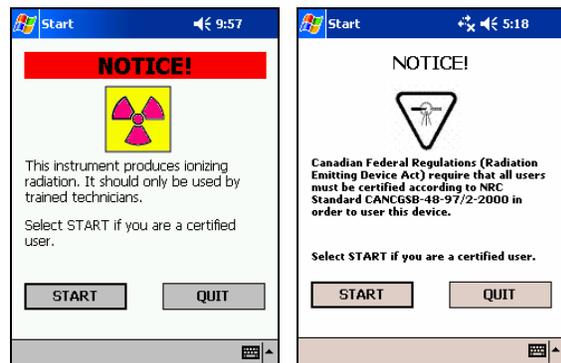
### 3.4 PERFORMING A TEST FOLLOWING APPROPRIATE RADIATION SAFETY PROCEDURES

#### Starting the Analyzer:

When an operator opens the Innov-X software on the iPAQ, he or she will be presented with one of the displays shown to the right. Provided an operator has received training from an authorized Innov-X trainer, he/she should tap the START button to begin using the analyzer.

**In Canada, INNOV-X ANALYZERS MUST BE OPERATED BY CERTIFIED USERS ONLY!**

From this point the operator is presented with the main menu of the analyzer to choose an operating mode and begin testing (described in Chapter 4). The remainder of this section is dedicated to operational and safety aspects that pertain to safe use and storage of the analyzer.



### Starting a test using the trigger.

When the trigger is depressed, the analyzer supplies power to the x-ray tube and opens the shutter to emit x-rays.

If deadman trigger is enabled, the trigger must be depressed for the duration of the test. Releasing the trigger will close the shutter and immediately end the test. If deadman trigger is disabled, pulling the trigger once will start a test, pulling it again will stop it.



**Figure 3.1** Handle of analyzer. Trigger is located at top of handle.

### Starting a Test Using the “Start” Icon on the iPAQ Screen

This feature is disabled in all units shipped. It will become active only if the “deadman” trigger is disabled.

An operator may also begin a test by pressing the **Start** button on the touch screen, as shown at the right. The **Start** button, rather than the trigger, is generally used when the analyzer is docked into the testing stand.

This Feature is not available in Canada. All tests must be started via the trigger.



## 3.5 CORRECT AND INCORRECT INSTRUMENT USAGE:

The Innov-X XRF analyzer can be used in several different testing configurations. Safety guidelines are described for each configuration.

### Configuration 1: Usage as a Handheld Alloy Analyzer:

In this configuration the analyzer is held in the hand, placed on various types of samples and a test is performed. Samples include pipes, valves, large pieces of scrap metal, basically any sample large enough to be tested in place, rather than held in the operator’s hand. Point the instrument at a metal sample such that no part of your body including hands and/or fingers is near the aperture of the analyzer where x-rays are emitted.

Using the analyzer in this manner assures that the operator will not obtain a radiation dose to any body part or extremity in excess of naturally occurring background radiation. The radiation at any surface of the analyzer is < 0.1 mR/hr except at the exit port and the immediate area around the exit port.

The user should take care that personnel are not located within 3’ (1 m) of the front end of the analyzer during testing, in the direction of the x-ray beam. Provided the analysis window is completely covered, there is virtually no radiation being emitted around the area of the sample. However, if a small component or curved surface is being analyzed, some radiation will be detectable.

## Configuration 2: Usage in the Testing Stand

Innov-X strongly recommends that testing small pieces or small samples (rod, fasteners, turnings, XRF sample cups, bagged samples, etc.) be analyzed using the Innov-X Testing Stand. This allows the sample to be placed onto the analysis window of the analyzer without requiring the sample to be held by the operator. See figure below titled “Testing Stand Operation.”

Note for Canadian Usage: The testing stand is not available for use in Canada at this time because it has not received regulatory approval yet. When an interlocked version of the testing stand has received regulatory approval, it will be available for sale into Canada. Please contact Innov-X Systems for an update on this process at 781-938-5005.

**Figure 3.2** Testing Stand Operation. Please refer to **Section 2.7: Testing Stand** for assembly instructions.

**Warning:** Innov-X strongly recommends that operators do NOT hold samples in their hand for testing. Never hold a small sample in your hand, and test that sample, such that your hand is exposed to the x-ray beam being emitted from the analyzer. This type of testing produces a small but non-negligible radiation dose to the operator’s hand. Please see **Section 3.7: Radiation Doses for Several Scenarios** for dose levels. Also, see **Figure 3.4** for an example of incorrect usage.



**Figure 3.2.**

## Testing of Small Components:

Operators often are required to test small components, particularly in the field of alloy analysis. Examples of small samples include turnings, weld rod, wires, fasteners, nuts and/or bolts.

There are specific procedures to test small components. These procedures should be followed at all times. **Never hold a small part with your fingers or in the palm of your hand and perform a test. Doing so may deliver a significant dose of radiation to your fingers or hand.** Please refer to the Examples of Mis-use below.

Method 1: Testing a sample lying on a flat surface.



Figures 3.2.: Performing a testing for a sample lying on the surface of a table. This is a good way to test small samples, rather than holding them in your hand.

To analyzer small sample:

- Place the sample onto a flat surface.
- Place the window of the analyzer onto the sample and begin the test.

Safety Precautions:

Do not test samples in this manner at a desk or table where the operator is sitting. If the desk is made of wood or another non-metallic material, some radiation will penetrate the desk and may provide exposure to legs or feet if the operator is sitting at the desk or table.

Analytical Precautions:

If the sample does not completely cover the window, be sure the surface used does not contain metals or even trace levels of metals, as this may affect the accuracy of the XRF result. The XRF may report the presence of additional metals in the surface material. For this type of testing, it is good to place the sample onto a piece of 1100series aluminum alloy and perform the analysis. The operator should disable the aluminum analysis capability (See Section 8.3.3 in the manual for instructions).

Method 2: Use the testing stand as described above (see also Fig. 3.2).

### ***Examples of Incorrect and Possible Unsafe Operation:***

#### ***Improper Operation, DO NOT TEST SAMPLES LIKE THIS:***

Exposure to the operator's hand/fingers will likely be minimal for this type of a testing, because the operator's hands and fingers are not in the primary beam. However, Innov-X believes that this type of the analyzer sets a poor safety precedent in that any operation where the operator places their fingers or hands near the window should not be permitted.



**Figure 3.3.** Incorrect Usage. While the dose to the operator's fingers/hand is negligible, testing this way sets a poor safety example for other operators, possibly encouraging other unsafe usage. Innov-X strongly recommends against this type of testing.

**DO NOT TEST SAMPLES LIKE THIS:**

Never hold a sample in your hand such that any part of your body or appendages are exposed to the x-ray beam. Testing samples in this way may generate significant radiation exposure (up to 27 R/hr) to the operator's fingers.



**Figure 4.4** Extreme example of incorrect usage. An operator should NEVER hold small samples by hand

## 3.6 RADIATION WARNING LIGHTS AND LABELING:

### 3.6.1 Main Power switch and Indicator Light:

The main power switch is found on the rear of the unit and is shown in the figure to the left. Pressing the switch for several seconds will turn on the main power. A green LED indicates the main power is on. The main power must be turned on in order to operate the unit however, this switch DOES NOT turn on the x-ray tube. No power will be supplied to the x-ray tube unit the Innov-X software is started.



### 3.6.2 Probe Light and Probe Label:

The Innov-X analyzer is equipped with warning lights that alert the operator when the tube is receiving power, and when x-rays are being emitted from the analyzer. Please see Fig. 3.5.

When the red light on the front nose of the analyzer is ON continuously (not blinking), this indicates the x-ray tube is receiving a low level of electrical power and the shutter is closed. The system is producing a low level of x-rays internally in this condition, but the shutter is providing adequate shielding to keep x-ray levels below levels of detection. The instrument is safe to be carried around or set down in this configuration.

When the red light is blinking, this indicates the tube is powered, the shutter is open and the analyzer is emitting x-ray radiation out of the analysis window. The analyzer should only be pointed at a sample, or be in the testing stand with a sample resting on the window, in this configuration.

### 3.6.3 Display on Back of Analyzer:

The display on the back of the analyzer, shown in Fig. 3.6, provides a “testing” message to indicate that the x-ray tube is energized and the shutter is open. This display is for testing conditions (i.e. overhead) where the operator cannot see the Probe Light or the iPAQ display.

### 3.6.4 Label Behind iPAQ:

The analyzer also has a label just below the iPAQ indicating, as shown in Figure 3.7:

**CAUTION: Radiation. This Equipment Produces Radiation When Energized.**

This label is required by most regulatory agencies. The term “When Energized” refers to the condition where the tube is fully energized and the shutter is open. This condition is also indicated by the red blinking light on the probe.



**Figure 3.5.** Probe light and labeling. When the light is on continuously, the x-ray tube is receiving minimal power and it is producing a minimum level of x-rays. The shutter is also closed so there is no radiation exposure to the operator or bystanders.



**Figure 3.6.** Back light on analyzer.



**Figure 3.7.** Label behind iPAQ. Top version is used in Canada

### 3.7 RADIATION LEVELS FROM ANALYZER

Two pictures of the analyzer are shown below. In the first picture, all the relevant components referenced in this radiation safety section are displayed and labeled. The second picture shows a close-up of the front end of the window. The four sides A, B, C and D are indicated on this picture because they are referenced in terms of radiation levels output by the analyzer. The measured radiation levels for standard operating conditions are shown in the figures and tables below. Standard operating conditions are tube voltage operating at 35 kV, tube current of 5 uA, and 2 mm aluminum filtration.



**Figure 3.8** Innov-X Analyzer, Side View



**Figure 3.9** Innov-X Analyzer, Front View

Radiation Levels (mrem/hr) for Alloy Analysis, Standard Beam Conditions: 35kV, 5 uA, 2mm aluminum filtering:

Sample at Window	Trigger	Location A (Top)	Location B (Right Side)	Location C (Bottom)	Location D (Left Side)
Blank (Air)	<0.1	<0.1	<0.1	<0.1	<0.1
Metal	<0.1	<0.1	<0.1	<0.1	<0.1

**Table 3.1.** Dose rates (units of mrem/hr) at various locations with a metal sample covering the window and with no sample present. For “no sample” the analyzer is shooting the x-ray beam into air.

As shown in the Table 3-1, the dose to the operator’s hand is negligible. The radiation levels at the side surfaces of the instrument snout (aluminum housing) are all <0.1 mrem/hour. Despite these low levels of radiation, there is no reason for any body part to be in the locations denoted A, B, C and D!

Table 3-2 shows the radiation levels directly in the x-ray beam that is emitted from the analyzer. Radiation levels at the exit aperture (or “port”) are substantial. There is no reason for the operator or any personnel to be exposed by the direct beam. Operators should never hold samples in their fingers or cupped in their hands, as this may generate a significant radiation exposure.

Operations should never point the analyzer at another person and start a test, as this may also provide significant exposure to the person if they are within a few inches of the port of the instrument.

Radiation Levels in the Primary Beam Versus Distance from Port:

For Alloy Analysis, Standard Beam Conditions: 35kV, 5 uA, 2mm aluminum filtering:

Tube Conditions	At Trigger, or any part of operator’s body.	At Window	4 inches	12 inches	36 inches	48 inches
35 kV, 5 uA, 2 mm Al filtering	<0.05	28,160	2,080	186	24	14
15 kV, 25 uA, thinner filter material	< 0.05	27,780	1,620	145	19	11

**Table 3.2.** Dose rates (units of mrem/hr) in the direct x-ray beam being emitted from the analyzer

### 3.8 RADIATION DOSES FOR SEVERAL SCENARIOS

In this section we provide data, concrete examples of use and misuse of the analyzer and common questions and answers we encounter when training personnel on the safe use of the Innov-X analyzer. The goal is to explain scenarios of safe versus improper usage of the analyzer.

The table below presents radiation doses for normal operating conditions and also for examples of misuse of the analyzer and even extreme misuse. Innov-X provides installation training that includes detailed radiation safety training and documentation designed to prevent misuse of the analyzer

Example of Instrument Usage	Radiation Exposure and Comments
<p><b>Normal Operation - Dose to Hand:</b> User analyzes samples according to standard operating procedures described in this manual. Assumption: Operator using system with x-ray tube ON for 8 hours/day, 5 days/week, 50 weeks/year. (Practically constant usage).</p> <p><b>Normal Operation – Dose to Torso:</b> Analyzer is used under the same operating conditions described above.</p>	<p>Maximum exposure is to operator’s hand, at the trigger. Exposure is &lt; 0.1 mrem/hr. Annual exposure to hand is then &lt; 200 mrem (2mSv).</p> <p>US: Maximum exposure under OSHA regulations is 50,000 mrem annually. Thus continuous operation provides a dose that is at least 250 times lower than maximum allowed by OSHA.</p> <p>Canada: Maximum exposure under ICRP regulations is 500 mSv for radiation workers and 50 mSv for the general public. Thus continuous operation provides a dosage 250 times lower for a radiation worker and 25 times lower for the general public.</p> <p>Exposure to Torso is so low it cannot be measured. To be conservative we use the same figure as the trigger, &lt;0.1 mrem hr. Annual exposure using operating conditions above is &lt; 40 mrem. (0.4 mSv)</p> <p>Maximum allowed is 5,000 mrem under OSHA and 20 mSv under ICRP for radiation workers (1 mSv for general public).</p>
<p>For the x-ray energy emitted by portable XRF analyzers (10-60 keV region), the bone in the fingers will absorb radiation about 3-5 times more than soft tissue, so the bone would be at an elevated radiation risk compared to soft tissue. For this reason no person shall hold a test specimen in front of the window with the fingers in the direct beam, or direct the beam at any part of the human body. Reference: Health Physics 66(4):463-471;1994.</p>	
<p><b>Misuse Example 1:</b> Operator holds samples in front of window with fingers, such that fingers are directly in the primary beam. Do not do this!.</p>	<p>For fingers at the port, in the primary beam, the maximum dose to the fingers is 28,160 mrem/hr. Assume an operator performs a 10 sec test (typical). The dose to the operator’s fingers or hand is 28,160 x (10/3600) = 78 mrem. If the operator did this 641 times/year they would exceed the allowable annual dose of 50,000 mrem to an extremity. In Canada, the maximum allowed dose is 500 mSv/year (Canada ICRP radiation worker) or 50 mSv/year (Canada ICRP general public).</p> <p>If the test time was 30 seconds instead of 10 seconds, the operator would receive a dose of 234 mrem for each exposure, and thus would exceed the annual safe limit of 50,000 mrem after 213 tests.</p> <p>Even though it is unlikely to make this mistake so many times in a year, do not even do it once. Take the extra time to test a sample on a surface or use a testing stand. <b>Note: If the operator takes an average of only two shortcuts per week and places his/her fingers within the primary x-ray beam at the window, they will exceed the annual dose rate.</b></p>

**Misuse Example 2:**

Operator places analyzer against body and pulls the trigger to start a test. Analyzer tests to preset testing time (usually 10 seconds) unless operator pulls trigger again to stop test. This applies to analyzer being in contact with operator or with bystander.

Dose at exit of sampling window is 28,160 mrem/hr.

Dose for a 10 second exposure with analyzer in contact with Torso: 78 mrem (.78 mSv).

US: If an operator did this act 64 times in a year, the operator would exceed the annual safe dosage to the torso of 5,000 mrem/year. The maximum dose of 5,000 mrem/year is a whole body limit, which does not truly apply in this case because the x-ray beam size is small (about 2 cm<sup>2</sup> area – 1.5 cm x 1.3 cm – at the port). Applying correction factors for the beam size is complex and beyond the scope of this manual. The important point is that for proper operation there is no reason to ever expose any part of the human body directly to the x-ray source. This example serves to provide estimated exposure in the event this occurs.

If the testing time was 30 seconds instead of 10 seconds, thus the operator placed the port against his body or that of a bystander and performed a 30 second test, the dose would be 234 mrem. This is about the same as a mammogram. Repeating this gross mis-use 22 times would exceed the annual allowable limits.

Canada: Radiation worker would have to repeat this example (234 mrem exposure) of gross misuse 8 times to achieve the ICRP level of 20 mSv. (general public 1.3 times to achieve limit of 1mSv)

**Misuse Example 3:**

Operator manages to initiate a test for 10 seconds and exposes a bystander that is standing 12” away from analyzer port. What is exposure to bystander?

Dose to bystander at 1 foot is 350 mrem/hr. For a 10 second exposure dose is 1 mrem. This is 5,000 times lower than the allowable dose to a worker in a year. This would have to happen 5,000 times to for that worker or bystander to obtain the maximum allowable dose.

Note: The proximity sensor would automatically shut down the x-ray tube after 2 seconds, so this is an extremely improbable occurrence.

Formula for calculating other scenarios:

$$Dose = 1\ mrem \left( \frac{13.25}{D + 1.25} \right)^2 \times \left( \frac{t}{10} \right)$$

D = distance from port in inches  
T = testing time

Note 2: Equations to scale these to other scenarios involving longer or shorter tests, and bystander being at distances other than 12” are provided at right.

Example: Bystander is 3’ away from port for a 30 second test. In this case the dose is calculated as:

$$Dose = 1\ mrem \left( \frac{13.25}{36 + 1.25} \right)^2 \times \left( \frac{30}{10} \right) = 0.38\ mrem$$

US OSHA: Maximum allowable level is 5,000 mrem assuming bystander’s torso is exposed. Thus, this misuse would have to occur 12,500 times in a year to the same bystander before that bystander achieved his maximum allowed dose.

ICRP: 5000 times for rad worker, 250 for general public

### Comparative: Radiation Doses from Typical Exposures to Ionizing Radiation

Common medical and/or dental x-rays:	20-30 mrem each.
Mammogram:	100-200 mrem
Flying in a commercial jet coast to coast (6 hrs.):	1-2 mrem.
Daily exposure from background radiation: * depends on geographic location	0.3 to 0.5 mrem/day

**Table 3.3** Radiation Doses from Typical Exposures to Ionizing Radiation

From the above table, a single case of analyzer misuse, thus producing a one-time exposure of 70-250 mrem, is comparable with single-event common medical x-ray procedures such as an annual chest x-ray or mammogram, or 25-50 airline flights in a year, and thus is not considered harmful. Regular misuse, such as taking safety shortcuts twice weekly, produces radiation exposure that greatly exceeds these typical levels and should be avoided entirely.

## 3.9 COMMON QUESTIONS AND ANSWERS REGARDING RADIATION SAFETY

**Question:** When I'm shooting a piece of pipe or valve on a rack or on a table top, is there any exposure to people standing in other locations, or standing several feet away from the analyzer?

Answer: Even a thin amount of metal sample (1-2 mm thickness) is enough to completely attenuate the x-ray beam emitted from the Innov-X analyzer. Shooting a piece of material that covers the sampling window on the analyzer will completely shield any bystanders from radiation exposure. However, good practice recommends that the area for at least 4-5 feet in front of the analyzer is clear of people.

**Question:** If I forgot to switch the safety on the trigger to "ON", I pick up the analyzer and accidentally pull the trigger, is that dangerous to nearby personnel?

Answer: No, this example of misuse is not dangerous, but it may produce a non-negligible radiation exposure to nearby personnel. For an exposure to occur, the following things must happen. First, you must be holding the analyzer so that a bystander is actually standing in the x-ray beam being emitted. Just being near the analyzer is totally safe otherwise. Second, the bystander must be within 1-3 feet from the nose of the analyzer in addition to being in the beam path, to receive any appreciable dose. If all of these conditions are true, the dose received by a bystander is still extremely low. It ranges between 0.1 to 0.5 mrem depending on the exact location of the bystander. This dose is 10,000 to 50,000 times less than the allowed dose. Please see Misuse Example 4 in the table above.

**Question:** Do I need to create restricted areas where I am using the analyzer?

Answer: No, provided you are following normal operating procedures there is no reason to restrict access to an area where the analyzer is in use. The operator should take precautions to keep any personnel more than 3 feet away from the sampling window of the analyzer in the event of accidental misuse as detailed above. Should the operator also elect to test small components like weld rod as shown in Figure 3.3, the operator should also be sure that no personnel are standing within about 4-5 feet of the sampling window.

**Question:** How does the x-ray tube in the Innov-X system compare to a radiography system used for taking images of metal parts.

Answer: The x-ray tube used in the Innov-X system produces between 1,000 and 10,000 times lower power than most radiography systems (0.5-1 watt for Innov-X versus kW for radiography systems). This is because a portable XRF is designed to perform surface analysis of alloys and other samples, whereas radiography systems are designed to shoot x-rays entirely through metal components in order to obtain an image on the other side of the object being bombarded with x-rays. For example, many tube-based radiography systems use a 300-400 kV tube and currents in the tens or hundreds of milliamps (mA). The Innov-X analyzer uses a tube operating at 35 kV and 5-30 micro-amps. The radiation levels produced are therefore thousands or tens of thousands times lower with the Innov-X system.

**Question:** Should we use dosimeter badges with the Innov-X analyzer.

Answer: Dosimeter badges are required by some states, and optional by other states. Innov-X recommends that operators wear badges, at least for the first year of operation, as a general precaution to flag any misuse of the analyzer. Dosimeter badges are available for the torso (generally worn on the belt loop or shirt pocket) and are available as “ring” badges. The best single badge to obtain is a ring badge that is worn on a finger, on the opposite hand used to hold the analyzer. This will record accidental exposure for the most likely case – an operator grabbing a small sample and holding it in one hand while analyzing it. Note: these badges generally have a threshold of 10 mrem, and are renewed monthly. So it will take several cases of misuse even to obtain a reading on a typical badge. When purchasing a badge, obtain the type used for x-ray and low energy gamma ray radiation.

### 3.10 SAFE GUARDS AND EMERGENCY RESPONSE

The main safeguards to use as an owner of an Innov-X portable XRF are really intended to restrict access to properly trained operators. **Note: Canadian regulations require certified personnel to use the device, refer to section 3.0 in this chapter.**

1. Keep the system in a controlled location, where only authorized users are likely to have access to the analyzer at any given time.
2. Make a simple sign that is kept with the analyzer indicating that an operator must have completed a training class provided by your company or must have attended an Innov-X training course in order to use the analyzer. Note that when the Innov-X system is turned on, the screen displays a message indicating that the system should only be used by authorized personnel.

#### **Emergency Response:**

Because the Innov-X system is a battery operated, x-ray tube based analyzer, the emergency response plan is very simple. If the operator believes the analyzer is locked up in an “OPEN” position, they should do two things:

1. Press the On/Off switch on the base to power the analyzer off. The green LED indicator will turn off, indicating system power is off. At this point it is not possible for the analyzer to be producing x-rays.
2. As an additional precaution, the operator may remove the battery trap door at the bottom of the analyzer (have the nose pointing away from personnel), and pull out the battery. Even if the operator has failed to properly power the system off in Step #1, removing the battery guarantees that no x-rays can be produced. There is no electrical power being provided to the x-ray tube.

Note: It would be highly unusual for an operator to somehow lock up the analyzer with the x-ray tube powered on. This would require the operator to crash the iPAQ during an analysis. If this happens the analyzer will shut off the x-ray tube 10 seconds after the last communication with the iPAQ. However, if at any time the operator believes the x-ray tube is on and no test is in progress, powering off the analyzer and

restarting will automatically shut down the x-ray tube and close the shutter. It will no longer be possible to produce x-rays at this point.

### 3.11 DOSIMETER BADGES

Dosimeter badges are provided as a monthly service by several companies, listed in this section (see below). The badges are generally provided monthly, and the operator returns the previous month badges to the company for analysis. The operator receives a monthly report showing any personnel with readings higher than typical background radiation.

Dosimeter badges are required by some states, and optional by other states. Innov-X recommends that operators wear badges, at least for the first year of operation, as a general precaution to flag any misuse of the analyzer. Dosimeter badges are available for the torso (generally worn on the belt loop or shirt pocket) and are available as “ring” badges. The best single badge to obtain is a ring badge that is worn on a finger, on the opposite hand used to hold the analyzer. This will record accidental exposure for the most likely case – an operator grabbing a small sample and holding it in one hand while analyzing it. Note: these badges generally have a threshold of 10 mrem, and are renewed monthly. So it will take several cases of misuse even to obtain a reading on a typical badge. When purchasing a badge, obtain the type used for x-ray and low energy gamma ray radiation.

#### Dosimeter Companies:

Here are two companies that provide badges as a regular service. There are certainly many more.

Landauer Inc.  
Glenwood, IL  
708-755-7000

AEIL  
Houston, TX  
713-790-9719

### 3.12 TYPICAL REGISTRATION REQUIREMENTS

Innov-X maintains a database of the registration requirements for every state, including sample registration forms. Most states require some form of registration, and generally they require the registration to be received within 30 days of receipt of the instrument. Some states require no registration, while a few require notification in advance. Please contact Innov-X for specific questions regarding the state where the instrument will be used, or for copies of registration forms.

In general a company will have to provide the following information regarding the device:

1. Purpose of device. Generally this is “Analytical” or “Industrial.” Be sure to inform the state registration office that the device will NOT be used for radiography or for medical uses.
2. Radiation Safety Officer – Monitors training, safe use, and controls access to the instrument.
3. Authorized Users – Trained by Innov-X Factory Authorized Representatives in the safe and proper use of the XRF.
4. Operating parameters of the analyzer – 35 kV, 5-30 micro-amps.
5. Type of system, either fixed, mobile or portable. Generally the correct choice is “Portable.”
6. User Training Specified – Indicate that only individuals receiving manufacturer training, documented by a manufacturer’s training certificate will operate the instrument.
7. Personal Monitoring. This may be required by radiation control authorities. Many registration forms will ask that you indicate whether or not you intend to perform dosimeter monitoring.
8. Copy of Registration & Manual at the Job Site

If you have any questions regarding the type of registration form or filling out the form, please contact Innov-X Systems. Many states may confuse a portable XRF system that uses a tube with medical or

industrial radiography systems. This is because of the relative newness of portable tube-based systems. In all likelihood, Innov-X personnel have experience providing the necessary documentation to the state in question, and can readily assist the customer in this process.

# Chapter 4 Operation

## 4.0 OPERATION - GENERAL

Power to the instrument is controlled by the ON/OFF button located at the rear of the analyzer. The green LED next to this button will illuminate when the analyzer power is on. The iPAQ operates on the Microsoft Windows CE ® operating system and is activated separately by the power button on the right top face, just over the display. The trigger is locked via the software.

## 4.1 WORKING WITH THE HP iPAQ Pocket PC®

The Microsoft Windows CE ® operating system and Innov-X software provided on the iPAQ handheld computer are operated by user input through the touch screen. For comprehensive details on the iPAQ's operation, please refer to the iPAQ reference materials included with your unit.

### General tips

- The Start Menu is found in the upper left corner of the iPAQ screen. This is used to launch all applications, including the Innov-X Systems Analyzer software.
- The instrument is designed as a “point and shoot” system that requires little, if any, entry of information for most operations. In the event the user modifies the grade library, enters testing information data, or performs other functions, it will be necessary to enter data via the virtual keyboard, which can be accessed by tapping the keyboard icon in the lower right corner. The iPAQ also includes character recognition software. This can be selected from the drop-down menu to the right of the keyboard icon.
- The File toolbar which will be used to Change Functions, Screens and Options is located at the bottom of the screen.
- It is possible to cut, copy, rename and delete files from within Windows File Explorer by selecting the file to be modified and holding the stylus on the screen for 2 seconds.
- Pressing buttons on the bottom of the iPAQ will perform various functions that are described in the iPAQ documentation. The button on the right hand side of the analyzer is the iPAQ task manager. Pressing this button will show all programs that are currently open. Open files can be closed from this menu. Simply hold the stylus on the file for a few seconds. The option to close the file will appear.

## 4.2 OPERATION - MAIN SOFTWARE SCREENS

The Innov-X Software consists of three main screens:

- **Main Menu screen:** Used to select the analysis mode, open the results screen, and change the administrator password.
- **Analysis Screen:** Used to change settings, edit libraries, and perform tests.
- **Results Screen:** Displays results from current reading, allows scrolling back to previous test results. Allows recorded data to be exported to a comma delimited file which is directly compatible with Microsoft Excel.

### 4.2.1 Innov-X Main Menu

The main menu below appears upon startup. The Main Menu allows you to choose an analysis mode, as well as perform certain administrative functions such as changing your login password. The modes which

are available on the analyzer are shown in blue. For information on adding additional analysis modes to an analyzer, please contact the Innov-X Sales Department at 781-938-5005.

- **Use the Main Menu to select the desired analysis mode.**  
The analysis mode can be selected by either tapping on the name of the method (shown in blue) or by selecting the appropriate mode from the Modes menu.
- The administrative password can be changed by selecting **Options** → **Change Password**.
- It is possible to go directly to the Results Screen by selecting **View**→**Results**. If the results screen is opened in this manner, it is possible to view results when the iPAQ is not connected to the analyzer.

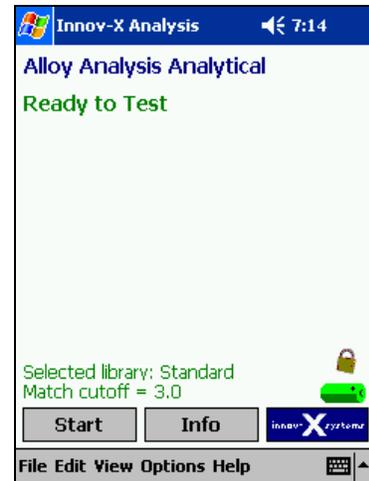


## 4.2.2 The Analysis Screen

Selecting a mode opens the analysis window for that mode. All data acquisition and analyzer control are done from this window. This window allows the user to start or stop an analysis, change testing parameters, and modify the fingerprint and grade libraries (Alloy Analysis only).

The analysis screen runs continually while during normal instrument operation. From the results menu, it is always possible to go back to the Analysis screen by selecting **File**→**Exit** or by tapping the X in the upper right hand corner of the screen.

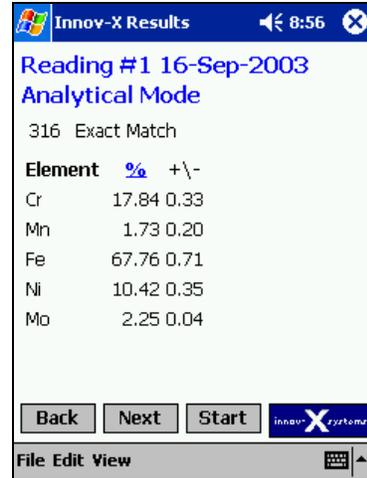
The analysis screen for Analytical mode is shown to the right. Screens from other modes are similar and will be described in later in this manual. The analysis screen shows the name of the mode that is currently active, a start/stop button (which is inactive in most cases), an info button that is used to enter descriptive information for any given test, a trigger lock and a battery indicator. In addition, a message appears directly below the name of the mode which will indicate the current state of the analyzer. Typically it reads “Ready to Test,” but also provides other information in certain circumstances. Any mode specific information will be displayed at the bottom of the screen above the menu choices.



## 4.2.3 The Results Screen

The Results screen displays the current reading and old data. All data handling functions such as exporting and deleting readings are carried out from this screen. Once the Results Screen is open, the user may start new tests without going back to the analysis screen by pulling and holding the trigger. Tapping the X in the upper right hand corner will return the user to the analysis screen without starting a test. If no analysis mode is running, an Exit button will appear which will close the Results screen.

The Results screen is automatically shown at the completion of any analysis. It can also be accessed from the analysis screen for any mode or the **Main Menu**, by selecting **View**→**Results**. Once the Results screen has been opened, the information which is displayed can be changed by selecting options from the View menu. The various viewing options will be described in detail in later chapters.



### 4.3 PASSWORDS - ABOUT PASSWORD PROTECTION

Certain functions such as adding and deleting fingerprints from the libraries, and Pass/Fail setup have been specified as Administrative Level Functions. These functions are described in detail in later sections of the manual. In order to use these functions, a password must be entered. The default password is set as the lowercase letter “z”. This password can be entered whenever the system prompts for a password.

#### Changing the Administrator Password.

The Administrator password may be changed at any time from the **Innov-X Main Menu** by choosing **Options**→**Change Password**. When the change password option is selected, this screen will appear.

If you are changing the password for the first time, enter the letter “z”; otherwise enter the current system password. Then, choose a password and enter it twice, once in the “New Password” box and again in the “Confirm Password” box. Passwords may be any combination of letters or numbers.



### 4.4 STANDARDIZATION

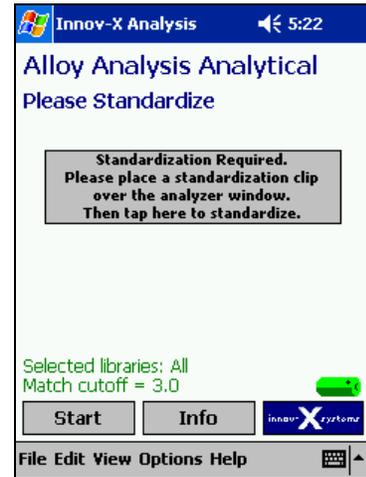
#### 4.4.1 Standardization Procedure

Before performing tests, it is necessary to standardize the instrument. This automated procedure involves collecting a spectrum on a known standard (Alloy 316) and comparing a variety of parameters to values stored when the instrument was calibrated at the factory. If there are any problems with the instrument, they will be indicated by an error message.

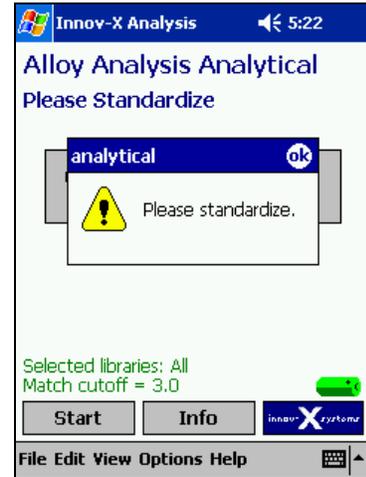
The standardization procedure takes about 1 minute. Standardization must be done any time the analyzer hardware is initiated or restarted and must be repeated if the instrument is operating for more than 4 hours.

It is possible to re-standardize the instrument at any point while the software is running. Standardization is always initiated from the Analysis Screen of any Mode.

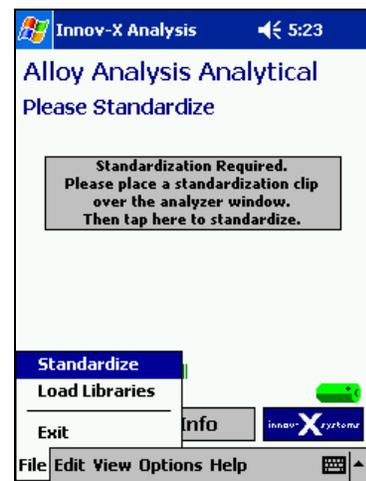
If the analyzer is restarted, you will be required to standardize the instrument before performing any measurements. This is indicated by the message “**Standardization Required. Please place a standardization clip over the analyzer window. Then tap here to standardize.**” on the analysis screen



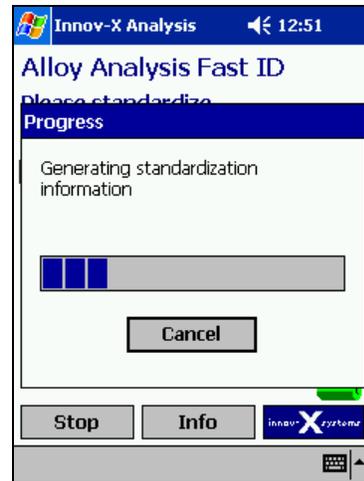
It is not possible to start a test before standardization. If the trigger is pulled before the standardization procedure is completed, a message box will appear. Press **ok** to acknowledge and clear the message.



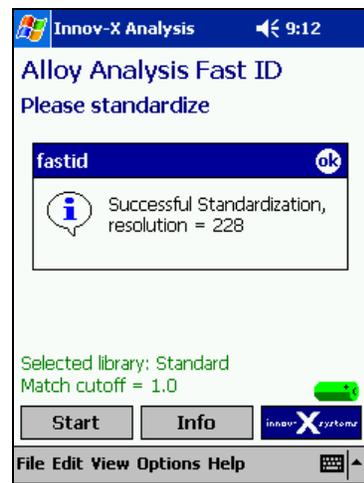
To initiate the standardization procedure, snap the standardization piece on the front of the instrument. Verify that it completely covers the analyzer window. When using a standardization mask with a weld collimator, be sure that the solid portion of the mask covers the analyzer window. Tap the grey box in the center of the screen or select **File**→**Standardize** to begin.



When standardization is in progress, the red light on the top of the instrument will blink, indicating that the X-ray tube is energized and the shutter is open. In addition, a status bar will appear, tracking the progress of the measurement.

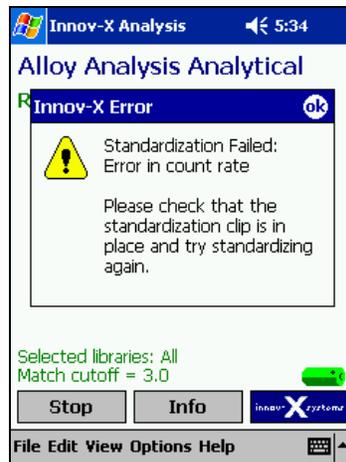
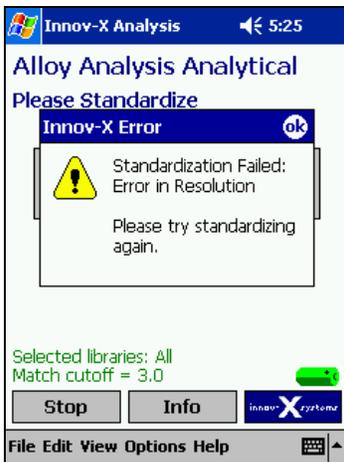


When standardization is complete, the message “Successful Standardization” will appear, along with the resolution of the instrument. Tap **ok** to acknowledge and clear the message. The instrument is ready for testing.

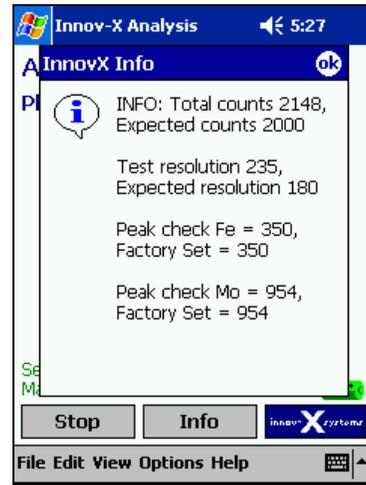
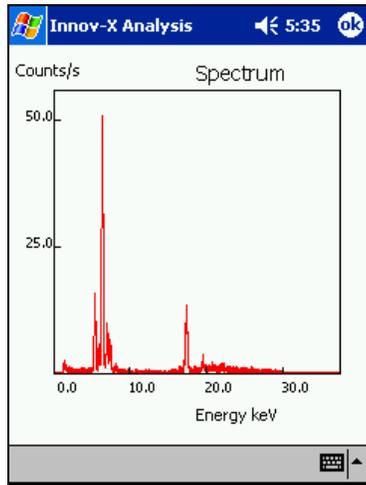


#### 4.4.2 Standardization Errors

The analyzer performs several diagnostic checks during the standardization process. If the standardization fails, the instrument will prompt the user regarding the next step. Several errors could occur while standardizing: “Wrong Standardization Material,” “Error in Resolution” or “Error in Count Rate”

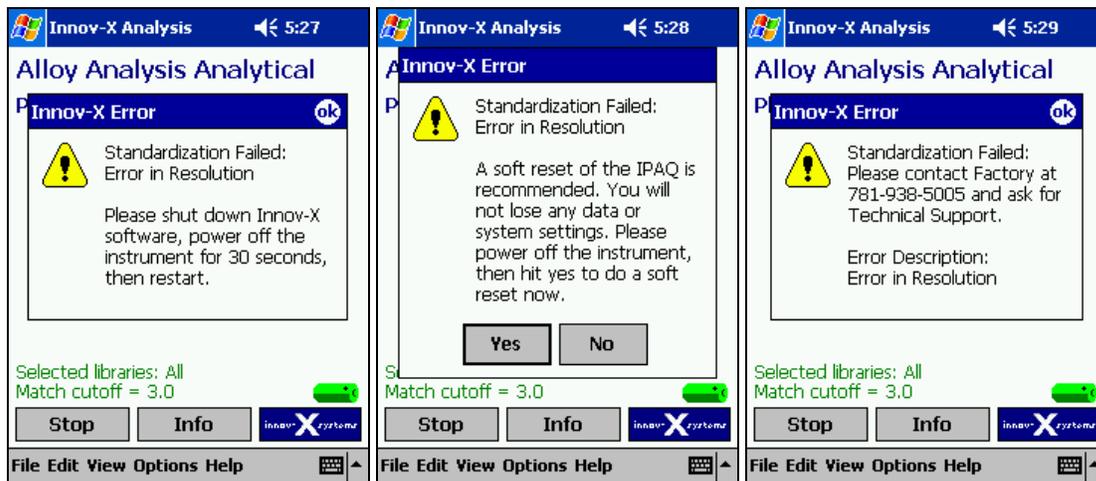


After closing the Standardization Failed message, two additional screens will appear. The first is a picture of the spectrum generated during the standardization. The second is a summary comparing factory set values for resolution, count rate, and peak positions to values calculated during the standardization.



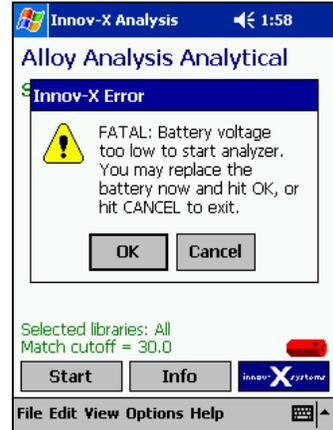
When standardization fails, verify that the standardization mask is in place, and attempt standardization again. To restandardize after a failure, tap the grey box in the center of the display, or choose **File**→**Standardize**. If you are using a weld collimator, make sure that the solid part of the mask is covering the window.

If standardization fails again, exit the analysis screen and power off the instrument. Restart and restandardize. If the standardization fails a 3<sup>rd</sup> time, you will be prompted to perform a soft reset of the iPAQ. Selecting Yes on this screen will automatically soft reset the IPAQ. You should also power cycle the instrument. Restart and restandardize. If the standardization fails again, replace the battery in the instrument and attempt another standardization. If this fails, please contact the Innov-X Systems service center at **781-938-5005**.



### 4.4.3 Battery Replacement and Initialization/Standardization

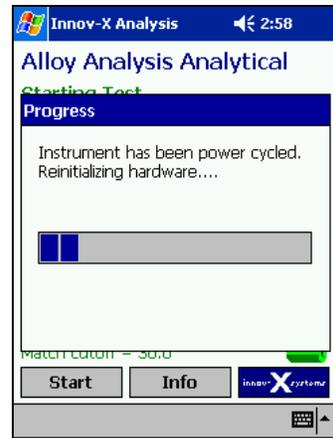
When the battery is too low to take a measurement, an error message will appear:



In order to continue testing, replace the battery immediately, and then tap “OK.” The analysis screen will remain open, and the instrument will reinitialize. This process will take 1 minute. It is not necessary to re-standardize, provided that less than 4 hours has elapsed since the last standardization and the battery swap is completed within 10 minutes.

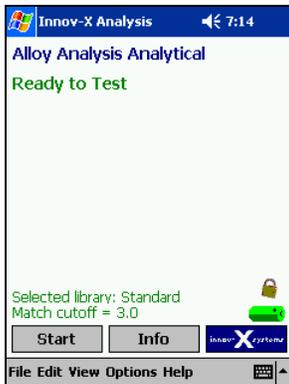
After re-initialization is completed, testing can continue.

If the battery is not replaced, and cancel is selected, the Analysis screen will close. When the software is restarted, the instrument will go through a complete 1 minute initialization and will require standardization.

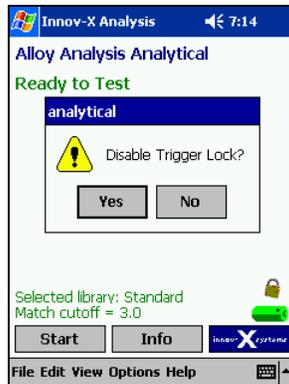


## 4.5 THE SOFTWARE TRIGGER LOCK

Innov-X analyzers are equipped with a software trigger lock which prevents the trigger from being actuated unintentionally. The lock is released by tapping an icon on the iPAQ screen. Once the lock is released, it will remain unlocked for subsequent tests, until more than five minutes has elapsed between tests. At that point, the trigger lock will be activated and will need to be disabled before additional testing can commence.



Tap the lock icon located directly above the battery indicator.



Select yes to disable the trigger lock



The open lock icon indicates when the trigger is disabled.

## 4.6 TEST INFORMATION - LABEL INPUT

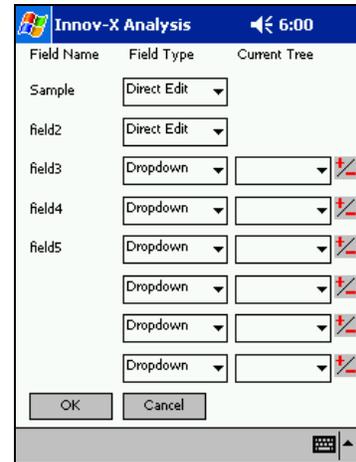
Information such as sample name, and identifying characteristics can be stored with each measurement. This is done from the test information (Test Info) screen which can be accessed from the **Analysis Screen** of any mode by tapping the **Info** button, or selecting *Edit*→*Edit Test Information*.

The **Test Info** screen consists of eight fields. The name and format of each field can be changed by using the **Modify Test Info Template** feature described in section 4.6.1 **Modifying the Test Info Template**. The process of entering test information prior to each analysis is described in section 4.6.2 **Entering Test Information**. Finally, the process of entering or changing test information after the analysis has been completed is described in section 4.6.3 **Editing Test Info from the Results Screen**.

### 4.6.1 Modifying the Test Info Template

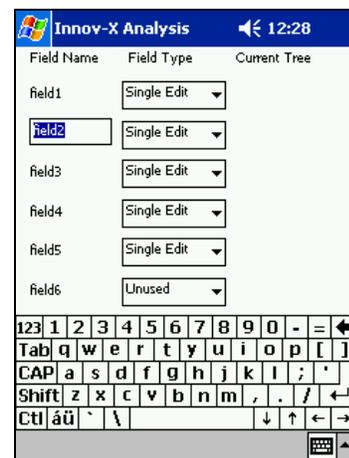
Test Info fields are modified via the Modify Test Info Template option found in the edit menu on the analysis screen in every software mode. Each field can be designated to be Direct Entry, Drop-down, or Tree. Direct entry fields allow users to enter characters directly from the virtual keyboard, or a bar code reader. Drop down menus provide a list of options to choose from. Trees are more complicated drop-downs; which allow users to subdivide large numbers of choices for ease in quickly locating the correct label. For example, a user may set up a tree with several parts for a main assembly. Subassemblies for the parts can be linked to their parent parts.

To make any changes to the Test Info format, select *Edit* → *Modify Test Info Template* from the analysis screen of any Mode. Modifications of Test Info screens are specific to each mode, and will need to be made to each mode if more than one is used.



#### 4.6.1a Changing Field Names

Field names can be edited by tapping on the current name. This will open an editable text box. A new name can be entered with the virtual keyboard. Selecting another cell or tapping **ok** will save this info.

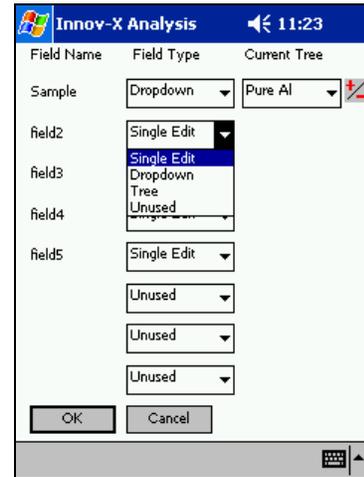


#### 4.6.1b Selecting Field Type

From the Modify Test Info screen, the type of field can be selected from a drop-down menu. Simply tap the arrow in the Field Type box for the field being modified.

- Select **Direct Edit** for a text field which will accept data from the virtual keyboard, or a bar code scanner.
- Select **Drop-down** for a drop-down list
- Select **Tree** for a Drop-down menu with many choices, some of which may be grouped into categories and subcategories.

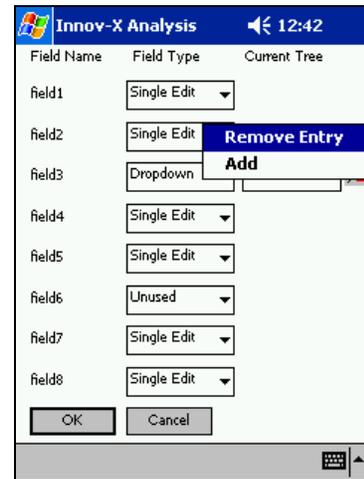
Select **Unused** to eliminate the field from the Test Info screen.



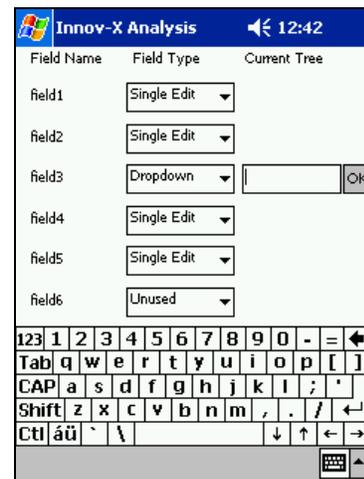
#### 4.6.1c Changing Drop-down Menu Entries

Once a field has been designated a drop-down menu, entries can be added or deleted by clicking the +/- symbol to the right of the field. Two choices will appear; **Remove Entry** and **Add**.

To delete a drop-down entry, first select the label to be deleted, then press +/- and tap **Remove Entry**.

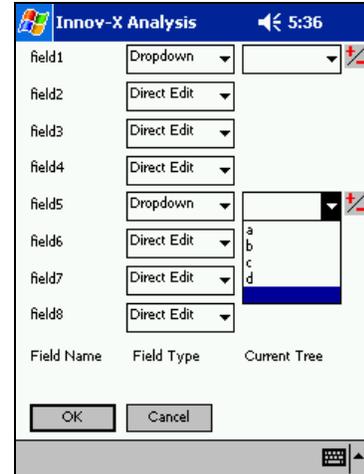


To add an entry from a drop-down list, tap the +/- symbol next to appropriate field, and select **Add**. Type the new info into the blank text box that appears. Select **OK** and the entry will be added to the drop-down menu.



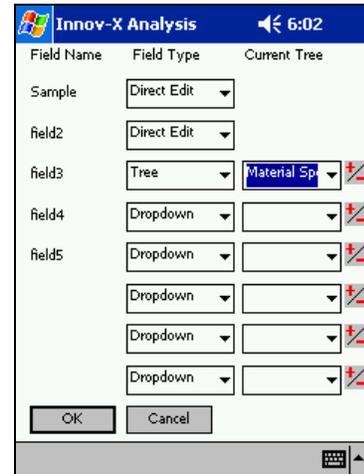
Repeat the process above to complete the complete drop-down list.

If it is anticipated that a drop-down field will not be used for all samples, enter an empty field as a choice so you can choose to leave the field blank.



#### 4.6.1d Changing Tree lists.

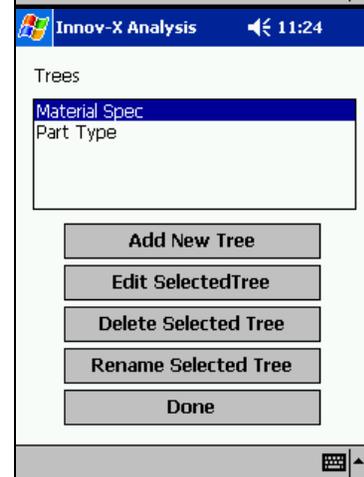
Once a field has been designated a tree, modifications to the contents of the tree can be made by tapping the +/- symbol to the right of the tree.



All modifications to trees are made from the menu shown on the right.

It is possible to add, edit, delete or rename trees. Select the appropriate choice from the menu to perform any of these functions.

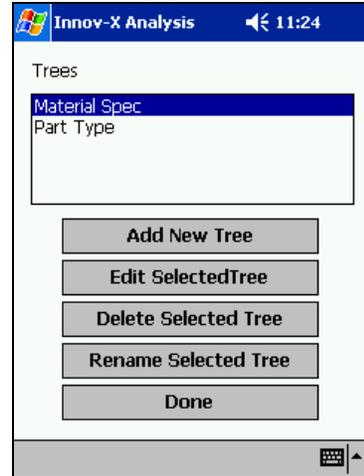
When you have finished creating/editing your tree, highlight it and select **Done**.



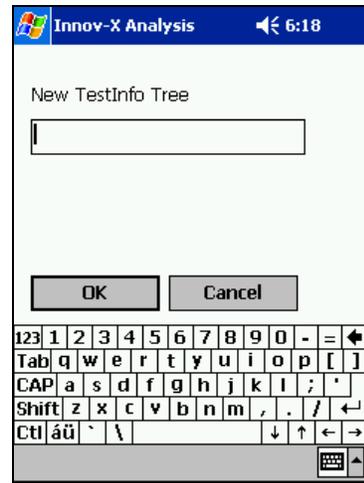
The following is an example of how a user might create a tree:  
 A manufacturer of tubes and valves tests all parts to ensure that they're made of the proper material. The company's QC procedure involves labeling each test with the part number of the item. Rather than forcing operators to look through a long list of part numbers, a tree is created in order to subdivide the parts number into groups based on part type.

The procedure for creating the tree is as follows:

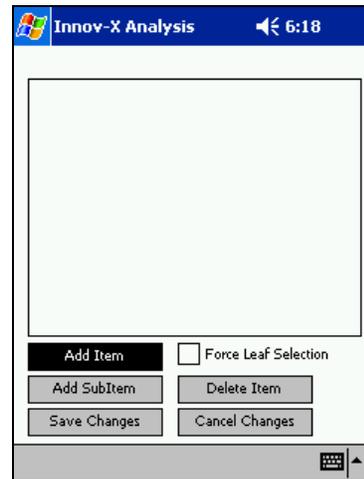
Select: Add New Tree:



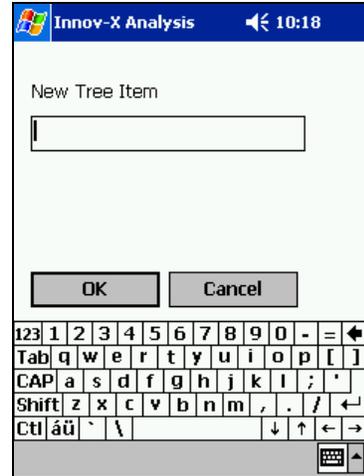
Enter the Name of the Tree in the text box and select OK.



Tap Add to add the first item

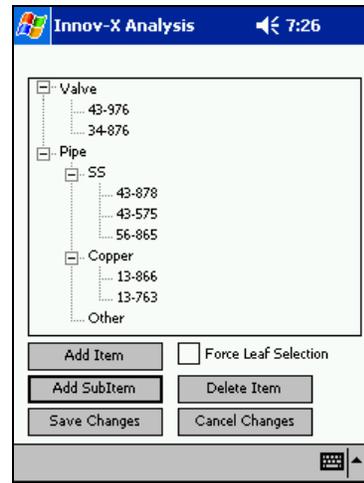


Enter the name of the item



Once the tree is started, continue to Tap Add Item to add a top level menu item, or select an item and tap Add SubItem to link a subcategory to the item. Continue until all items have been added.

In this example, the part numbers for pipes and valves are separated into categories. The pipes are further subdivided by material type.

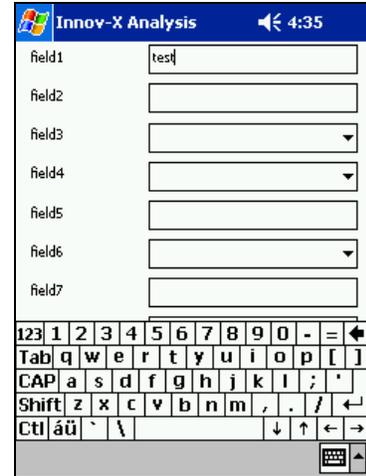


#### 4.6.2 Entering Test Information

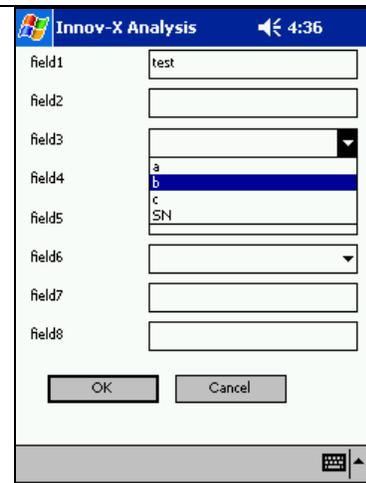
- To enter the Test Info screen, you must be in the Analysis Screen. If the Results Screen is open, tap the ⊗ in the upper right hand corner to return to the Analysis Screen. From the Analysis Screen, select *Edit*→*Edit Test Information*, or tap the **Info** icon.



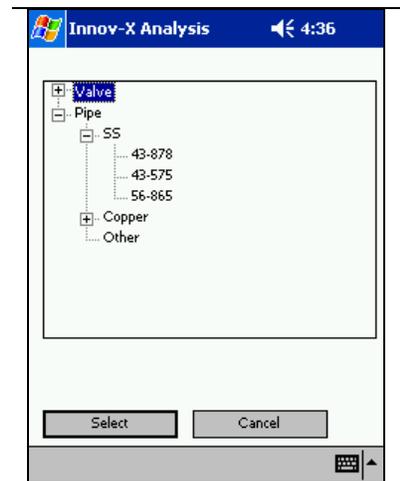
- To enter a unique sample name or number, select a direct entry field by tapping anywhere within the field. Use the virtual keyboard to enter the information.



- To select information from one of the drop-down menus, tap the arrow to the right of the box. Select the desired entry.



- Some drop-down fields are formatted as trees. To select information from these fields, tap the arrow to the right of the box. A screen will appear showing options. The plus (+) symbol will appear before some choices indicating the presence of sub-items. Tap on the + symbol to expand the menu. Tap on any item or sub-item to select it, then press **Select**.



- When all the necessary data have been entered, select OK
- The information entered in the test info screen will be saved with each reading until the test info screen is modified again.

### 4.6.3 Editing Test Info from the results screen

Test information can be edited, or added to a test after its completion.

- From the results screen, scroll to the reading to be modified.
- Select **View** → **Test Info** to see in the information which is already stored.
- Select **Edit** → **Edit Test Info** to bring up the editing menu.



You will then be presented with the same test information screen described in **Section 4.5.2: Entering Test Information**.

## 4.7 EXPORTING AND ERASING DATA

Because the memory of the iPAQ is limited, you should periodically backup the data on your analyzer, and erase the memory. Depending on test volume, it is recommended that all data is erased on a weekly or monthly basis.

### 4.7.1 Installing ActiveSync

In order to copy files between the iPAQ and a desktop PC, Microsoft Active Sync Software must be installed on the desktop PC. Innov-X strongly recommends that you download the latest version of ActiveSync from the internet. ActiveSync v3.7 may be downloaded from <http://www.microsoft.com/windowsmobile/resources/downloads/pocketpc/activesync37.msp>

If it is not possible to download the latest version, an ActiveSync CD (v3.5) was shipped with your analyzer. Check behind the foam in the instrument case.

The iPAQ cradle should be hooked up to the USB port on the desktop computer before installing software.

The Procedure for installing and setting up ActiveSync is as follows:

1. Insert the ActiveSync CD in your CD Drive. It will start automatically. The CD contains information about Getting Started with Your Pocket PC. This changes periodically, so it's difficult to describe exactly what the screens will look like. Step through the screens until you see the option "Install ActiveSync." Select this to start the installation process.
2. Follow the prompts on the screen. When given the choice, select "Run this program from its current location" and click OK.
3. Complete the install process. You will be required to restart your computer in order to complete the installation.
4. After restarting your computer, dock the iPAQ in the cradle. The iPAQ should automatically communicate with your computer. If it doesn't, check the connections and try removing the iPAQ and reseating it. If that doesn't work, try doing a soft reset on the iPAQ.
5. When the computer communicates, you will be prompted to "Set Up a Partnership." Select "Yes, with this computer"



6. Enter a name for your iPAQ and click next.



7. You will be prompted to “Select Synchronization Settings.” **Select “Files” only. It is important to make sure that Files is the only item checked. Otherwise, the files such as address books and emails will be copied from the desktop computer to the iPAQ.**



8. Step through the rest of the process.
9. A folder will automatically be created on the PC’s desktop with the name of the device entered in step 8 above. Results files saved on the iPAQ will automatically be synched and will be stored in this folder. Opening this folder and clicking on the name of the file will open the file in Excel.
10. After ActiveSync is set up correctly, copying results to a desktop computer will consist of

- a. Exporting results on the iPAQ. (described in section 4.6.2)
- b. Syncing the iPAQ to the computer
- c. Opening the results in Excel for viewing, or printing.

## 4.7.2 Exporting Results

All data from your Innov-X Systems analyzer can be exported as a comma delimited text file (csv). This format allows the data to be easily exported to spreadsheet programs. It is possible to export all data from a single day, or to export all data saved in the iPAQ. Results and spectra are exported separately.

To export or erase data, you must be in the Results Screen. This is automatically opened when a reading is taken, or can be accessed by choosing **View**→**Results** from any analysis screen.

From the results screen, select **File**→**Export Results**



You can choose to export All Readings or just Readings on a specific date. Choosing **All Readings**: will export all readings saved in memory and is a good choice if you want to backup all data stored on the instrument before deleting. If a large number of readings stored, this option will take several minutes.

Choosing **Export Readings on date** requires that you pick a date from the calendar below. It is strongly recommended that you use this option and export data on a daily basis.

The customize export option allows users with administrative password privileges to customize the format in which data is exported. This is described in **Section 4.7.3: Customizing Results Export**.



After choosing which readings to export, you may choose to export all data, or just data from a specific mode. Selecting the arrow to the right of the mode to export will open a drop-down menu. Select the mode for which you want to export data.

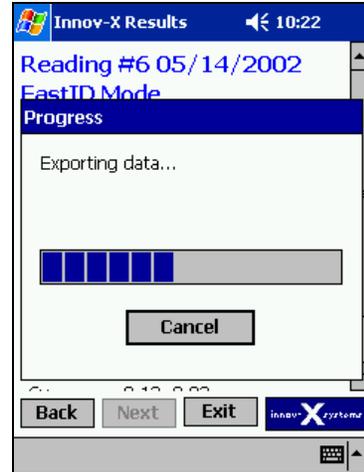
All standardization data are stored as results files. These data are automatically included in exported results files when the selected “Mode to export” is **All**. Additionally, it is possible to export only the standardization data by selecting **Standardization** as the “Mode to export.”

When the proper selections have been made, select **OK**. A **Save As** box will appear. Select the folder in which you want to save the data, and name the file. The file Type will always be **Comma Separated Values**. The recommended Location is Main memory and Folder is **None**. This will export files into the “My Documents” folder in the main Memory of the iPAQ.

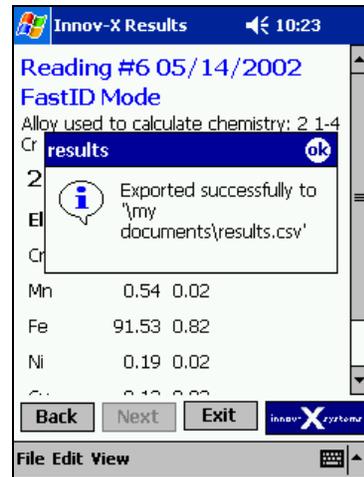
If you select a File Name which already exists, you will be asked if you want to replace the existing file. If you do, select **Yes**. Otherwise select **No** and choose another file name.



A status bar will indicate the progress of the export. It may take several minutes to export many readings. Daily downloading and weekly erasing of data simplifies and shortens this procedure.



When all readings are exported, a message will appear confirming the export. Tap **ok** to acknowledge and clear the reading.



### 4.7.3 Customizing Results Export

All units come with a standard results export format which reports a variety of information relevant to a test. Users can select which fields are exported as well as modify the order.

To modify exported results files, select **File** → **Export Readings** from the Results screen.

Tap the **Customize Export** box.

Enter the administrative level password when prompted.



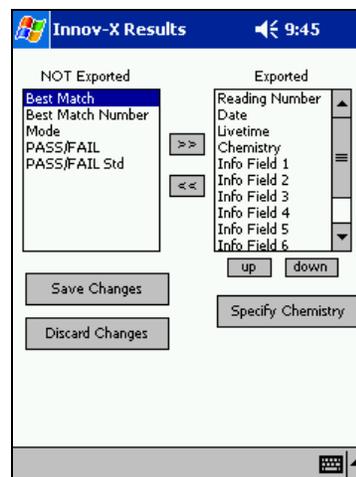
Two columns appear on the screen; the column on the left lists fields which will NOT be exported, and the right-hand column lists fields which will be exported.

Fields can be moved from one column to another via the >> and << buttons located in the center of the screen

Exported field order can be changed by using the **Up/Down** buttons. Select a field and move it up or down as desired

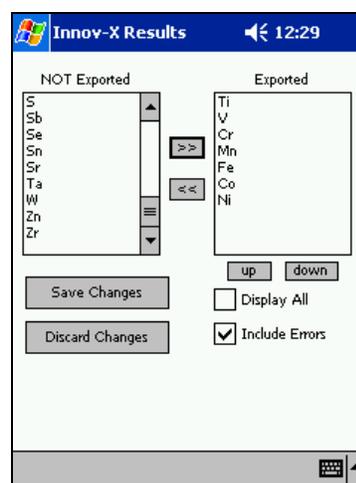
Once all changes have been made, choose **Specify Chemistry** if changes need to be made to the list of exported elements.

In chemistry is not edited, select **Save Changes** to keep the modified settings, or **Discard Changes** to ignore any changes.



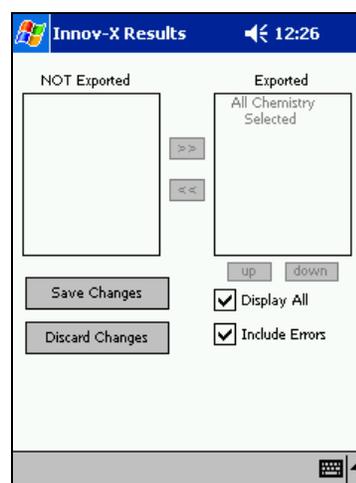
The Specify Chemistry screen resembles the previous screen. Move elements to the appropriate column, depending on whether or not an element should appear in exported files.

Select **Include Errors** to export the error associated with each measurement.



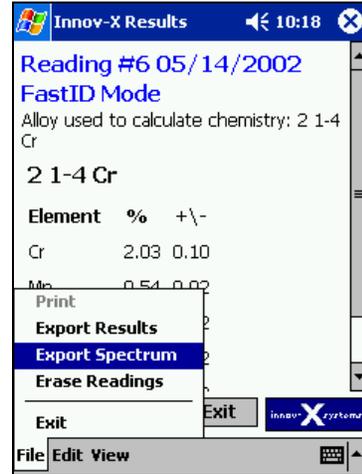
Select **Display All** to include all measured elements. *This setting is recommended, as it will ensure that all data measured with the instrument is exported.*

When all changes have been made, tap **Save Changes** or **Discard changes**, depending on whether the changes should be saved.

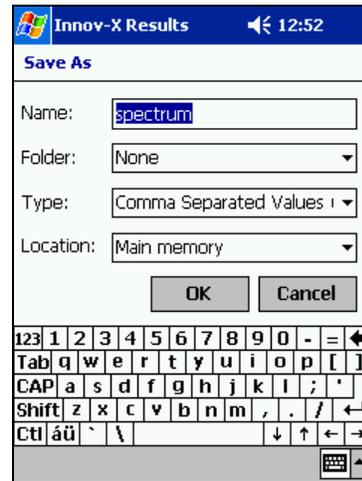


## 4.7.4 Exporting spectra

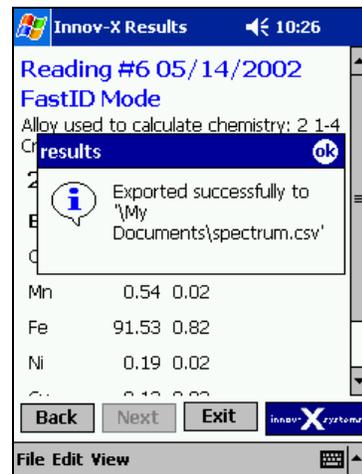
Only one spectrum may be exported at a time. In the results screen, scroll to the reading for which you wish to export the spectrum, and **Select File**→**Export Spectrum**.



Choose the File name, and make sure that **Comma Separated Values** and **Main Memory** are selected. This will save the spectrum to the My Documents folder in the Main Memory of the iPAQ.



A message will appear indicating a successful export. Tap **ok** to acknowledge and clear the window.



## 4.7.5 Erasing readings

It is possible to erase a single reading, a range of readings, all readings from a specific data, or all readings before a specific date.

In order to erase a single reading, the reading to be erased must be displayed on the screen before selecting delete. If necessary scroll to the reading you wish to delete.

In order to select a range of readings, you must have a reading open from the date you wish to delete the readings. If a reading from the desired date is not open, you may select **View**→**Go to date**, and select the appropriate date.

The reading displayed in the results screen is not relevant if you want to delete all readings from a specific date, or all readings before a specific date .

From the results screen, select **File**→**Erase Readings**.

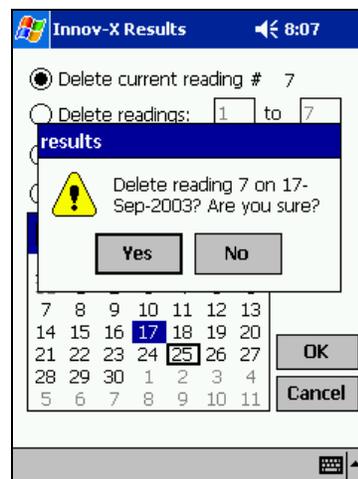
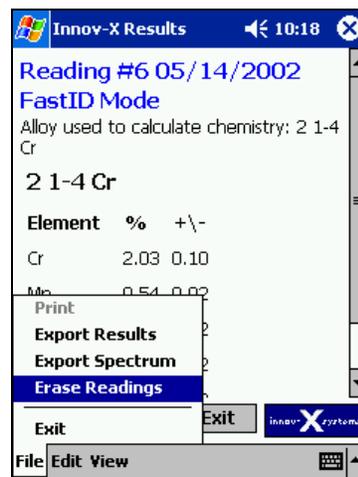
A message box will appear prompting you to enter your password. Enter your administrative level password and select **OK**.

A dialogue box will appear allowing a choice of which results to delete. Select the appropriate choice:

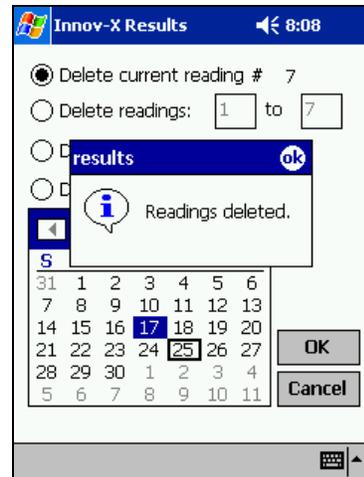
- Selecting **Delete current reading** will delete the reading that is currently open.
- Choosing **Delete readings XX to XX** will delete a range of readings from the date of the reading that is currently open.
- **Delete all readings on date** deletes all readings from a specific day.
- **Delete readings before date** deletes all readings taken prior to a specific day.

If you select **Delete all readings on date** or **Delete readings before date**, you must specify a date from the calendar. The default date is the current date.

When you've selected the readings to delete, Click **OK**. You will be asked if you're sure you want to proceed. If you want to proceed with the data erase, select **Yes**. Otherwise, click **No**.



A message will indicate the readings were successfully deleted. Tap **ok** to acknowledge and clear the message window.



# Chapter 5 Soil Analysis

The Innov-X analyzer can be used to analyze in situ (directly on the ground), bagged or prepared soil samples. A guide to Soil analysis using field portable X-ray fluorescence is found in the appendix. This document summarizes EPA Method 6200 which is the standard protocol for field screening. It also provides information on prepared sample testing.

## 5.0 CHECK STANDARDS

It is recommended that a check standard is measured after each standardization, and periodically throughout the day. Innov-X provides several NIST certified standards for verification. The certified values for these samples are provided in the appendix. At least one standard should be measured for a minimum of 1 minute. Elemental concentrations for elements of interest plus or minus the error on the reading should be within 20% of the standard value. The Field screening guide in the appendix describes in more detail recommended quality assurance considerations.

The standards provided with the XRF analyzer are contained in XRF sample cups with a Mylar window (through which the soil can be viewed) on one side, and a solid cap on the other side. Samples should be measured in the sample cup, through the Mylar window. The best way to measure a prepared sample is using the test stand. If this is not available, the sample may be placed on the ground, and the analyzer may be pointed downwards in full contact with the soil cup. Do not hold the soil cup in your hand while measuring.

## 5.1 SAMPLE PRESENTATION

### **In situ testing:**

In situ testing is performed by pointing the analyzer at the ground. Any grass or large rocks should be cleared away and the analyzer should be held such that the front of the probe head is held flush to the ground.

Since dirt can accumulate on the analyzer window, it is recommended that the window is wiped clean after each analysis. The window should also be checked to ensure it is not ripped or punctured. Instructions for replacing the window are found in the appendix.

### **Bagged or prepared sample testing:**

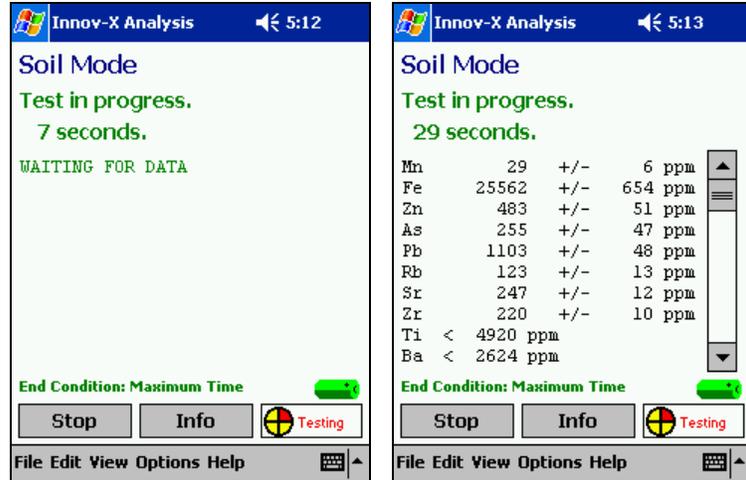
It is strongly recommended that all prepared samples be analyzed in the testing stand. Samples should be placed on top of the testing stand, completely covering the window. **Never hold prepared or bagged samples while testing**, as this could expose the operator to the x-ray beam.

Avoid measuring very thin samples, as this can affect results. Prepare samples cups to contain at least 0.5 inches of packed samples. When analyzing bagged samples, make sure that sufficient sample exists in the bag to completely cover the window with a sample thickness of a minimum of 0.5 inches.

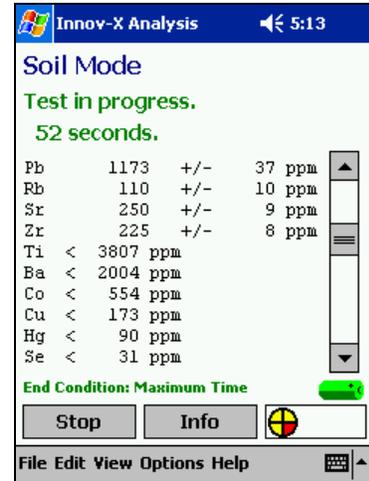
## 5.2 TESTING IN SOIL MODE

After the instrument has been standardized, testing can begin. Simply pull the trigger or press **Start** on the iPAQ screen to begin the test. The red warning light on the top of the instrument will blink, indicating X-rays are being emitted. The screen will display the words “Test in progress” and the time elapsed. The word “Testing” will blink on and off in the low right hand corner of the screen.

After a minimum time has elapsed, intermediate results will be displayed on the screen. Until this minimum time has elapsed, the words “WAITING FOR DATA” will appear instead. This minimum time can be set by the user by selecting *Options*→*Set Testing Times*, which is described in **Section 5.4: Soil Mode Options**. Each line of the results display shows the name of an element, its calculated concentration and the error on the measurement. This error is the 1 sigma error on the counting statistics of the measurement. The error will decrease with increased testing time.



Too many elements are measured in soil mode to display them at one time. However, it is possible to use the scroll bar located to the right of the chemistry display to view other elements. The complete display shows detected elements first, listed in order of emission line energy, from lowest to highest. Following the detected elements are the elements which are below the detection limit of the instrument. These elements are shown as less than a calculated LOD. This LOD is defined as three times the error on the counting statistics of the measurement.

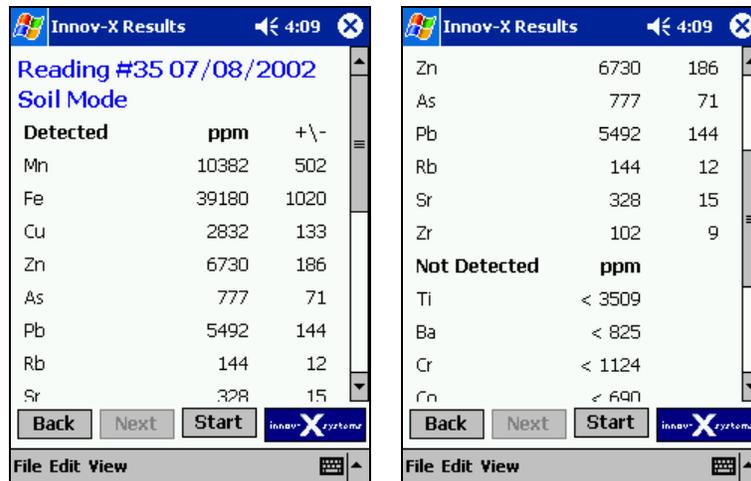


When the measurement is complete the results screen will open, displaying the final results of the measurement.

## 5.3 SOIL RESULTS SCREEN

### 5.3.1 Results View Menu

The standard Soil Mode results screen displays the concentration (in ppm) and error in measurement for detected elements, followed by the list of non-detected elements with the calculated limit of detection for each element for that test. If the display does not show soil chemistry results, change the display by selecting *View*→*Results*.

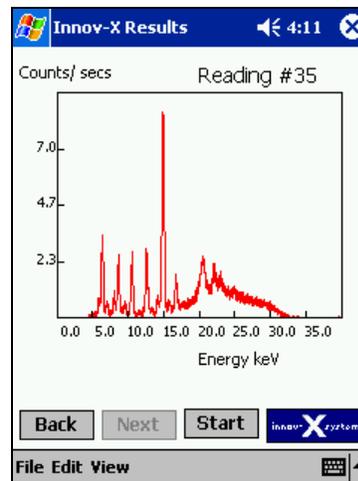


The standard soil chemistry display can be modified by using the View Menu. As with all Innov-X analytical modes, it is possible to view spectra and Test Information.

### 5.3.2 Spectrum Screen

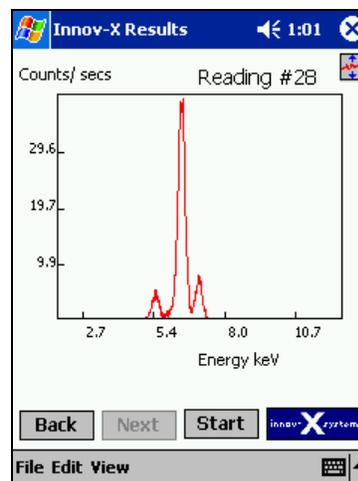
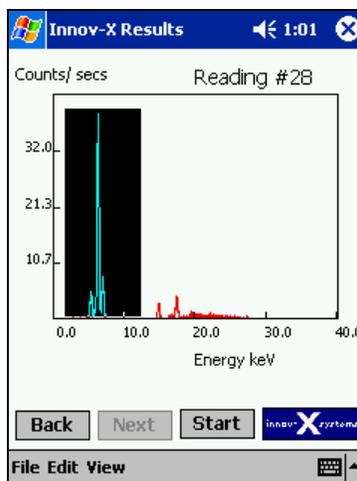
This screen displays a plot of the x-ray fluorescence spectrum for an individual test, plotting the intensity on the y-axis versus the energy of the fluorescence x-rays on the x-axis.

Tapping on the spectra will show the energy scale and counts rate at the selected point



It is possible to zoom in on certain areas of the graph by selecting one corner and drawing out the region

Tapping the symbol in the upper right hand corner beneath the X will restore the graph to full scale.



### 5.3.3 Test Info Screen

The test information screen shows any test information that was entered prior to the start of the test. Changes to that test information can be made by selecting **Edit**→**Test Information**.

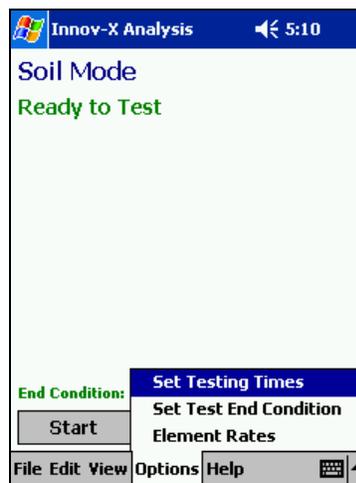
## 5.4 SOIL MODE OPTIONS

The length of tests in Soil Mode is user settable. Users may select a minimum testing time, and as well as choose from a variety of test end conditions.

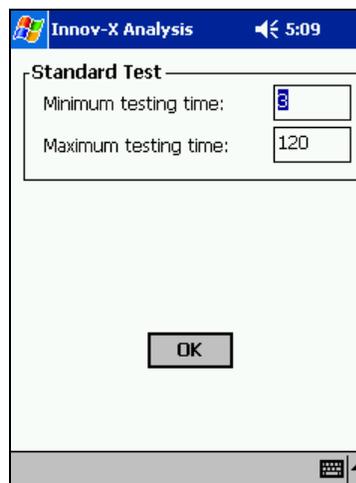
The options related to test time are contained in two menus: **Options**→**Set Testing Times**, and **Options**→**Set Test End Condition**. **Set Testing Times** contains minimum and maximum testing time information, while **Set Test End Condition** allows the user to select test end conditions.

## 5.4.1 Set Testing Times

To set the minimum and maximum test lengths, select **Options**→**Set Testing Times**



A screen appears prompting you to enter a Minimum and Maximum Testing times. Instruments equipped with the optional LEAP package will be able to set Light Element Testing times in this screen, as well.



The minimum testing time is the required time that must elapse before results can be calculated. Live Update results will not be displayed on the screen until the minimum has elapsed, likewise a test must complete the minimum time before any test end condition can be used. If a test is stopped before the minimum testing time has elapsed, the test will be aborted, and no results will be calculated.

Maximum testing time is relevant only if “Maximum Testing Time” is selected from **Set Test End Condition**. This will automatically end the test at a preset testing time. Typically, the maximum testing time will be in excess of 30 seconds, and may be 1 or 2 minutes, depending on detection limits and desired precision.

It should be noted, that all testing times in this section refer to “Real Time,” the time the measurement takes when timed on a normal clock. The time stored with each analytical result (accessible by selecting **View**→**Test Information** from the Results screen), refers to the test’s “Live Time”. This is the amount of time that the analyzer hardware was collecting spectra. Since there is some detector dead time associated with a measurement, the live time of a test will be slightly shorter than the preset “Real time”.

## 5.4.2 Soil Mode Test End Condition

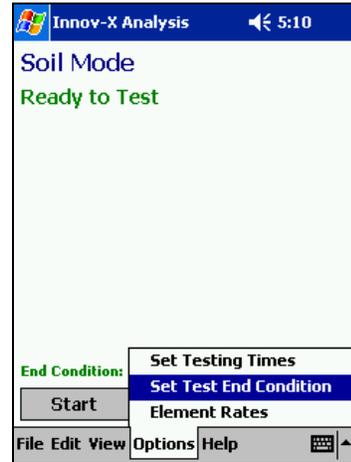
Four options exist for the test end criteria in soil mode. Depending on your application, you may choose to end the test manually, at a preset testing time, or when the uncertainty in the measurement is within a

specified relative standard deviation of the reading. Additionally, you can set up an action level for a single element. As soon as the measuring statistics are good enough to ensure that that the reading is above, below or at the action level, the test will end automatically. This allows for very rapid tests for elements that are well above or below an action level.

In all modes, pressing Stop, or pulling the trigger will end the test. If the minimum testing time has elapsed, results will be calculated. Otherwise the test will be aborted without calculating results.

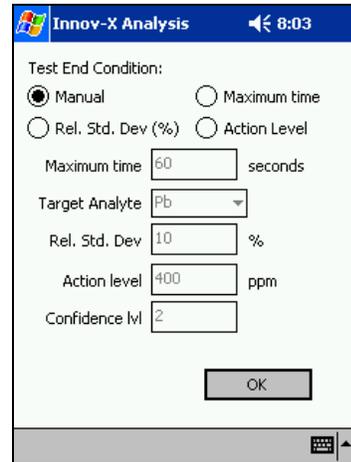
Changes to the test end condition are made by selecting **Options**→**Set Test End Condition**

The currently selected end condition will be displayed at the bottom of the screen above the Start button on the Ready To Test screen.



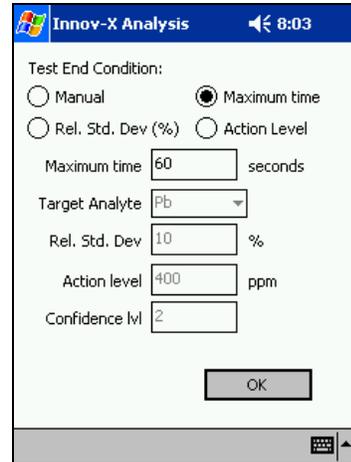
**Manual:** This option allows you to look at the results which are being continually updated on the screen and determine when the results look satisfactory. The test will continue until the trigger is pulled, or **Stop** is tapped on the iPAQ screen. Results will be calculated if the testing time has exceeded the Minimum Test time which is set up in **Options**→**Set Testing Times**. In order to preserve battery life, the software will stop if the testing time exceeds 300 seconds, since there is little to no advantage to continuing a test beyond 300 seconds.

To use Manual Test End Condition, simply choose **Options**→**Set Test End Condition** and select **Manual**. Press **OK** to return to the analysis screen.



**Maximum Time:** If Maximum Time is selected, the test will continue until the preset time is reached. This is useful if you wish to do a set of measurements with the same testing time.

To choose to end test based on a maximum time, select **Options**→**Set Test End Condition** and select **Maximum Time**. Enter the desired testing time in the appropriate box. Tap OK to save your selections.



**Action Level:** System ends test when result for target analyte including chosen precision level is above or below pre-set action level.

To choose to end a test based on an Action Level, select **Options**→**Set Test End Condition** and select **Action Level**. Select a target analyte, specify an action level in ppm, and a confidence level. This confidence level refers to the number of sigma required for the precision. This should typically be set to 2. Tap **OK** to save your selections.

Innov-X Analysis 8:03

Test End Condition:

Manual  Maximum time

Rel. Std. Dev (%)  Action Level

Maximum time 60 seconds

Target Analyte Pb

Rel. Std. Dev 10 %

Action level 400 ppm

Confidence lvl 2

OK

**Relative Standard Deviation (RSD):** When RSD is selected as a test end criteria, the system will end a test when the relative standard deviation on a target analyte reaches a pre-set level. This standard deviation is specified as a percentage of the reading. For example, if the measured value for an analyte was 1000 ppm, and the RSD was set to 10, the reading would stop when the error reached 100 ppm, or 10% of 1000.

To choose to end a test based on a Relative Standard Deviation, select **Options**→**Set Test End Condition** and select **Rel. Std. Dev (%)**. Select a target analyte and the desired Relative Standard Deviation. Tap **OK** to save your selections.

Innov-X Analysis 8:04

Test End Condition:

Manual  Maximum time

Rel. Std. Dev (%)  Action Level

Maximum time 60 seconds

Target Analyte Pb

Rel. Std. Dev 10 %

Action level 400 ppm

Confidence lvl 2

OK

## 5.5 LEAP Mode (Light Element Analysis Program):

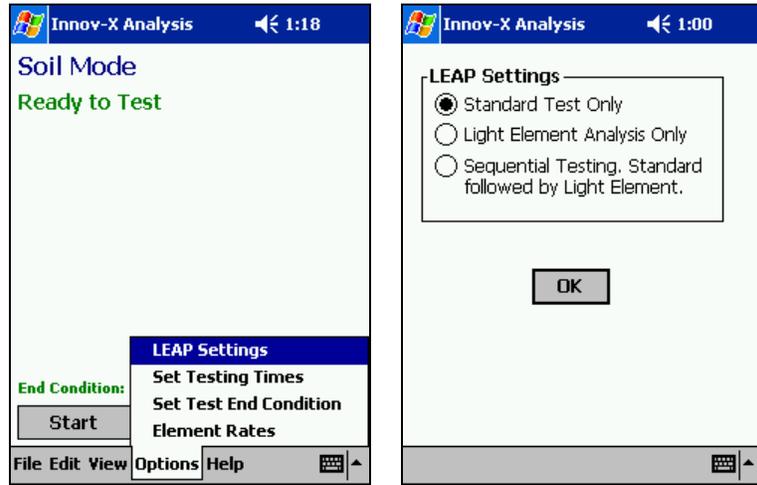
*This is a factory installed optional module. Instruments can be upgraded to LEAP capabilities. Please contact the Innov-X Systems Sales department for information and pricing.*

The LEAP module provides the lowest possible detection limits for elements lighter than iron. The standard LEAP package includes the elements Ti, Ba and Cr. Elements as low as Phosphorus can be detected with the Advanced LEAP package which includes a thin window detector.

The standard x-ray beam conditions used by Innov-X environmental analyzers are designed to provide good excitation for a wide range of detected elements. However it is not possible to select one beam condition which provides the absolute best excitation conditions for all elements of interest. Elements such as Chromium produce lower energy x-rays than other elements analyzed. These lower energy x-rays are not as effectively excited by the standard conditions. LEAP works by changing the X-ray tube beam conditions to settings which are optimized for the detection of elements lighter than iron. Instruments are factory calibrated with the LEAP beam conditions for all applicable elements.

## 5.5.1 LEAP Settings

To activate LEAP, select **Options**→**LEAP Settings** from the Soil analysis screen. This brings up the menu shown below on the right.

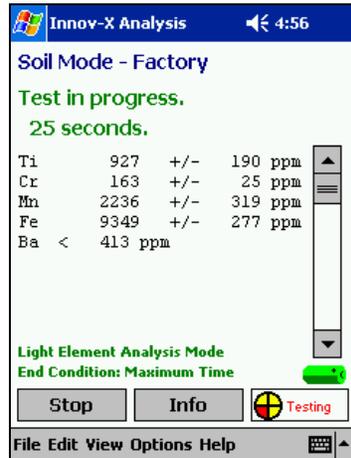


**Standard Test Only:** The analyzer will provide analysis for the standard suite of elements.

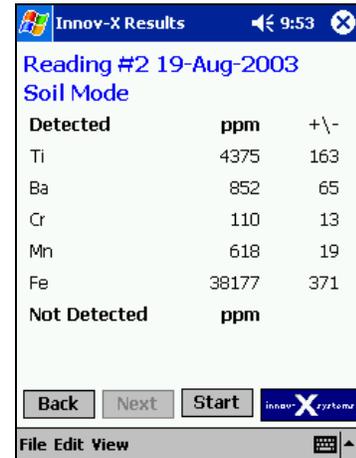
**Light Element Analysis Only:** The analyzer will provide analysis for elements in the LEAP suite (Typically Ti, Ba and Cr)

**Sequential Testing:** When sequential testing is selected, all tests will start with an analysis of elements in the standard suite. If that test ends due to reaching the selected end condition of Maximum Test Time, Action Level, or RSD, then the analyzer will immediately begin a second test analyzing the LEAP suite of elements. At the conclusion of this test, the Results screen will open with two new entries. The first summarizes the standard test results, while the second summarizes the LEAP results. For safety reasons, the second test will not begin if the test ends due to user intervention (pulling the trigger or hitting Stop). In this case, the Results screen will open with only one reading.

If Light Element Analysis Only is activated, the words “Light Element Analysis Mode” will appear above the currently selected End Condition. Instrument operation in this mode is identical to Standard (Non-LEAP) analysis. Tests can be started or stopped either by pulling the trigger, or by tapping the Start/Stop button on the iPAQ screen. The results screen for a test will show results for all elements analyzed with the LEAP mode.

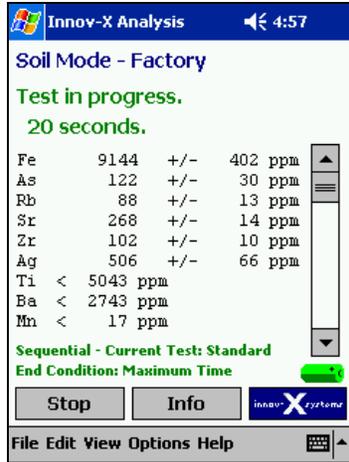


Test in progress screen, LEAP Only, Live Updates on

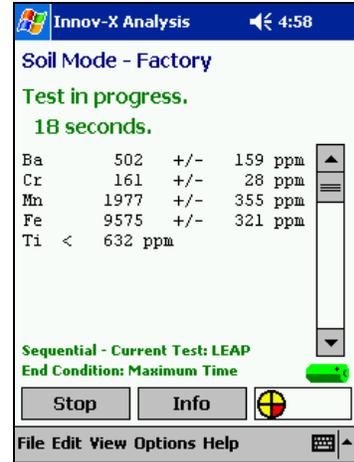


Results Screen Showing LEAP results

If Sequential testing is selected, the words “Sequential – Current Test: Standard” will appear above the currently selected End Condition. When a test is started, the instrument will appear to operate in the same manner as a Standard test. However, if the test ends according to the specified end condition (excluding Manual), the results screen will not open. Instead, the timer will reset to 0, and the description of the current test will change from “Standard” to “LEAP”. The live update screen will begin to show analysis for all LEAP elements.



Test in progress screen, Sequential.  
First Test – Standard Analysis.

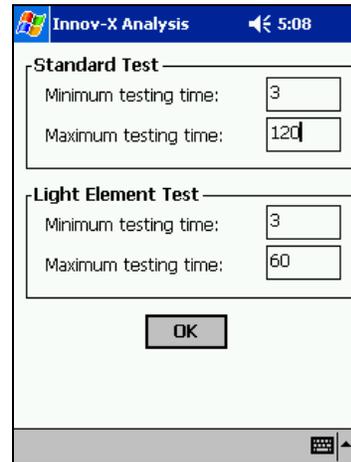


Test in progress screen, Sequential.  
Second Test – LEAP Analysis

### 5.5.2 Testing Times

To set the minimum and maximum test lengths for LEAP analysis, select **Options**→**Set Testing Times**.

The testing time screen includes an extra section labeled “Light Element Test” that is not found on non-LEAP systems. These are the minimum and maximum test lengths for any LEAP tests.



As with standard tests, the minimum testing time is the required time that must elapse before results can be calculated. Live Update results will not be displayed on the screen until the minimum has elapsed, likewise a test must complete the minimum time before any test end condition can be used. If a test is stopped before the minimum testing time has elapsed, the test will be aborted, and no results will be calculated.

## Appendix 1: Troubleshooting Guide—Soil Analysis

Problem	Possible Solutions
<p><b>Software won't start:</b></p> <p>Software will not start when the Innov-X Systems Icon is tapped.</p>	<p>The flash card or the iPAQ may not be correctly seated in the black external sleeve. Remove the flash card and press it firmly into its holder. Press the iPAQ down into the black sleeve.</p>
<p><b>Software won't start:</b></p> <p>Software doesn't start when the Innov-X System icon is tapped; instead, the following error message occurs: "Cannot find 'startup' (or one of its components). Make sure the path and filename are correct and all the required libraries are available"</p>	<p>The flash card or the iPAQ may not be correctly seated in the black external sleeve. Remove the flash card and press it firmly into its holder. Press the iPAQ down into the black sleeve.</p>
<p><b>iPAQ locks up:</b></p> <p>iPAQ screen "locks up" and doesn't respond when screen is tapped or buttons are pressed</p>	<p>Remove the iPAQ from the analyzer and perform a soft reset by pressing the tip of the stylus into the small indentation found on the bottom of the iPAQ. If the iPAQ is lying flat on a table with the screen facing upwards, the reset button is found to the extreme right of the side containing the power plug and connector.</p> <p>See Page 4 of the Compaq "Getting Started" manual for an illustration showing the location of the reset button.</p>

<p><b>Analyzer will not standardize</b></p>	<p>Try again. Choose File -&gt; Standardize to attempt a new standardization. Also be sure the standardization cap is on correctly, and that the solid half is in front of the window. It is OK to try this 2-3 times in the event of a failure.</p> <p>If a repeat attempt fails: Change the battery. In some cases the battery may be too low to provide enough power for tube startup. Follow this procedure:</p> <p>Reset the iPAQ;</p> <p>Turn off the analyzer and remove the battery.</p> <p>Verify that the battery is completely charged. If it is not, replace it with a fresh battery. Even if the battery has been recently recharged, remove it, and replace it in the analyzer.</p> <p>Restart the analyzer and software. Wait several minutes after the software has initialized before attempting standardization.</p>
<p><b>Results screen doesn't show new readings after a test is completed</b></p>	<p>Check the date on the iPAQ. The Innov-X Systems software indexes stored results by date. If the date is incorrect, results may not be displayed in the correct order.</p>
<p><b>Serial Communication Error Message:</b></p> <p>Serial Communication error occurs because iPAQ has been removed from instrument or cradle, with the software open and the instrument standardized.</p>	<p>This error reflects the temporary loss in communications when the iPAQ was removed. To avoid this problem, always use the <b>File / Exit</b> command to exit the software properly. Try simply removing and reseating the iPAQ to solve this problem. If that fails, see steps 1 – 4 below.</p>
<p><b>Serial communication error on startup, or while testing.</b></p>	<ol style="list-style-type: none"> <li>1. If the analysis screen is still open, attempt another test.</li> <li>2. Verify that the iPAQ is correctly seated in the analyzer by removing and replacing it.</li> <li>3. Remove the iPAQ and perform a soft reset. Replace iPAQ and restart software.</li> <li>4. Turn the analyzer off and restart it.</li> </ol>

<p><b>Results take a very long time to display on the first test of the day.</b></p>	<p>There may be too many readings stored in memory. Erase readings from the results screen by selecting <i>File</i> → <i>Delete Readings</i>.</p>
<p><b>Trigger will not start test.</b></p>	<p>Verify that the trigger lock is off.</p> <p>Reset the instrument. If this fails, call Innov-X Systems Technical Support at 781-938-5005.</p>
<p><b>Broken Kapton Window</b></p>	<p>The window is designed as a barrier to dust and dirt. If it is damaged, it should be replaced.</p> <p>To change the window:</p> <p>Turn off the analyzer</p> <p>Remove the screws holding the front plate in place.</p> <p>Remove the old kapton and adhesive, replace with new kapton and replace front plate.</p> <p><b>Important Note:</b> It is very important to avoid getting dirt and sharp objects within the probe, due to the close proximity of the detector. Do not use the analyzer without a kapton window for any length of time. Also, be very careful when removing/replacing screws in face plate so as to not accidentally damage the detector. If the detector is damaged, the instrument will require factory service.</p>
<p><b>Results screen shows message “Error in calculation: No Results”</b></p>	<p>The soil mode calculation is only valid for “soil-like” samples which contain primarily light elements such as carbon, oxygen and silicon. If a dense, highly metallic sample is analyzed, the calculation fails.</p> <p>Make sure the sample being analyzed is a soil sample, if it is and this message occurs repeatedly; call Innov-X technical support.</p>

## Appendix 2:

# **Metals in Soil Analysis Using Field Portable X-ray Fluorescence**

**A guideline to using portable XRF according to EPA Method 6200, basic overview of the technique of x-ray fluorescence (XRF), appropriate data quality assurance protocols and sample preparation steps for operators analyzing prepared soil samples.**

**Prepared by:**

**Innov-X Systems, Inc.  
January, 2003**

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## Section 1: Regulatory Status for Field Portable XRF

EPA Reference Method 6200 has been incorporated into SW486 under RCRA, and is now available for field portable XRF analysis of soils and sediments. Please call or email Innov-X Systems for a copy of Method 6200.

### **Method 6200: Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment.**

Features of this method:

1. It is a field screening method, for analysis of *in-situ* or bagged samples.
2. The method provides basic quality assurance methods, including calibration verification, determination of instrument precision, accuracy and limit of detection.
3. The method recognizes that some XRF instruments do not require site-specific calibrations by the operator, that is, the factory calibration provides appropriate data quality.
4. The method recommends that a minimum of 5-10% of samples tested by XRF be confirmed by an outside laboratory using a total-digestion EPA analytical reference method.

The purpose of EPA Method 6200 is NOT to replace laboratory analysis. There are two primary sources of error in assessing a site for metal concentration: **Analytical error** and **Sampling error**. Analytical error is the error in the analysis of any one sample by whatever technique is used, for example XRF, ICP, or AA. Sampling error arises when too few samples are collected and tested. In this case an incomplete picture of the extent of metals contamination may be obtained. Although any one sample may be analyzed with very high analytical accuracy, measuring too few samples may result in contamination plumes being mis-judged in size, or depth into the soil. In extreme cases contamination may be missed entirely.

EPA Method 6200 was developed to reduce Sampling Errors by increasing the number of samples measured. In general, a large number of screening-level measurements provide a better characterization of contamination than a small number of measurements produced by sample removal and analytical analysis. Portable XRF is an ideal tool to make a large quantity of measurements in a short period of time. A large number of in-situ samples provides detailed data on contamination profiles, depth (provided surface soil is moved aside), and approximate contamination levels. Portable XRF also can provide results with a high degree of analytical accuracy on any given sample. Please see Section 2 “Overview of Field Usage” for this discussion.

## Section 2: Overview of Field Usage:

Field portable XRF is generally used in three ways to test for metals in soil:

- ❑ **In-situ soil testing:** The XRF is placed directly onto the ground for soil testing. Operators remove any plant growth and foreign objects so that the analyzer probe is flush to the soil.
- ❑ **Bagged soil sample testing.** A soil sample is collected in a thin plastic bag (i.e. a “Baggie”) and testing directly through the Baggie. Except for a few elements – namely Cr, V and Ba – testing through the thin plastic used for a plastic bag has little effect on the test result. Results for Cr, V and Ba will be lower by 20-30%.
- ❑ **Prepared soil sample testing.** Prepared sample testing assures the operator of the maximum possible accuracy. Prepared sample tests require a sample to be collected, dried if necessary, sieved and ground into a powder. The prepared sample is then placed into a baggie or XRF cup for analysis. **A complete Soil Preparation Guide is provided in Appendix 1.**

All analytical methods require a uniform, homogenous sample for the best results. **XRF is no different!** The methods described in EPA Method 6200, namely In-situ and bagged sample testing, are considered *field-screening methods*. Although a field-screening method, in-situ testing is a valuable technique because it generates a great deal of data very quickly. Prepared soil samples generally offer the best accuracy, albeit with several minutes of sample preparation required per sample.



Figure 1. Use of a field portable XRF for in-situ soil testing.

### Subsection 2-A: Data Quality Objectives.

The objectives of the testing generally determine the mixture of in-situ versus prepared sample testing. It is important to understand your data quality objectives (DQO) in order to determine the appropriate mix of field screening and prepared sample testing.

In-situ testing usually provides only screening-level data quality. This is because analytical testing always requires a uniform, homogeneous sample matrix. A laboratory achieves this by digesting the sample into a hot acid before analysis. Testing directly on the ground does not ensure uniformity is met. Preparing a sample provides a uniform sample and likely better analytical data quality, although several minutes of testing time is required.

Most portable XRF operators use a mixture of in-situ and prepared sample testing. Several examples are described below. The exact mixture of in-situ and prepared sample testing depends upon the goals of the soil testing. The examples below serve as guidelines. Please contact Innov-X (1-866-4 Innov-X or 866-446-6689) to discuss your specific testing requirements.

**Example 1: Initial site investigation to provide detailed contamination data with efficient use of laboratory analysis costs.**

Problem: Site needs to be assessed for metals contamination. Little information is available about what metals are present, likely contamination levels or geographic profile of contamination.

The goal of testing is to determine what metals are present at what levels, both in area and in depth into soil. Additionally, testing will locate possible contamination plumes and/or possible sources of contamination.

**Recommended Testing Plan:** This example uses predominately in-situ testing. The analyst will perform in-situ testing, and gather samples into plastic bags for XRF analysis. A testing grid should be established in two or three dimensions, every several feet. XRF tests can be taken at each location or bagged samples can be collected from each location for later analysis. The in-situ data for each element analyzed may be plotted in a 2-dimensional grid (X, Y coordinates versus elemental concentration) to profile a site. These concentration profiles are ideal for showing contamination patterns, boundaries and plumes. Combining this data with historical use data from the site often allows the operator to deduce sources of contamination. Obtaining this level of geographic data with purely laboratory analysis would produce excessive analytical costs.

Prepared sample analysis should also be done to confirm the regions where in-situ data indicates low or non-detected levels of metal contaminant. There is little need to prepare areas where in-situ testing indicates high concentration levels. Innov-X recommends the same procedure as EPA Method 6200. For locations where in-situ tested indicate low or non-detected concentrations, calculate the total number of in-situ tests, collect 5% of this number of tests from the various locations, and prepare these samples according to Appendix 1. Use these prepared samples to confirm the findings of the in-situ testing. Send a subset of these prepared samples to a laboratory for confirmatory results.

Cost Justification. To adequately characterize a site may require 100-200 samples/acre to be sure the contaminated areas are firmly established. This work may be done with in-situ testing to generate laboratory savings of \$5,000 - \$10,000/acre depending upon the number of elements being analyzed. The cost reduction in off-site analysis often justifies the price of the XRF.

**Example 2: Monitor remediation efforts and assure site meets clearance levels before contractors leave the site.**

Goal: Minimize remediation costs by only treating contaminated soil, and obtain immediate verification that various site locations meet clearance objectives.

**Recommended Testing Plan:** This type of project uses a lot of both in-situ and prepared sample testing. Use in-situ testing to thoroughly delineate contamination regions in both area and depth. To determine depth profiles, test surface soil, remove at least 1-2 inches, and retest. Repeat this step as necessary to profile contamination depth to guide remediation activities. (XRF is a surface technique and only analysis the first few mm of soil sample). As part of clearance, collect several samples from “cleared” area. Prepare samples according to Appendix 1 and test with portable XRF.

If XRF indicates that concentration levels are in excess of clearance requirements, then continue remediation efforts.

If XRF indicates that concentration levels are below clearance requirements, then discontinue remediation efforts, and send a subset of the samples to an analytical laboratory to confirm results. Most operators safely assume that the cleanup requirements have been met for the elements in question, but await final analysis from the laboratory.

If XRF lists concentration levels as non-detected, but the detection level reported exceeds clearance requirements, send samples to a laboratory for final results.

**Cost Justification:** In-situ results are used to guide remediation efforts, in order to obtain maximum efficiency. Efficiency is produced because contamination boundaries are firmly established, thus avoiding remediation efforts with “clean” soil. Prepared sample testing is used to assure that clearance requirements are met on-site in near real-time (pending laboratory confirmation). Costs savings are generated by avoiding clearance failures. The contractors can leave the site earlier and will not be called back to the site for additional cleanup.

**Important Note:** Never clear a site based solely on in-situ testing. Always use well-prepared samples to make a clearance decision.

### **Example 3: Minimize volume of hazardous waste for treatment or disposal.**

**Goal:** For some cleanup projects, the cost of soil disposal in a hazardous waste landfill is much greater than disposal in a standard landfill. Testing soil samples with XRF may minimize the amount of “clean” soil that is inadvertently shipped to a hazardous-waste landfill.

**Recommended Testing Plan:** This example is almost entirely prepared sample testing. Representative samples are removed from the soil being hauled to landfill. Obtaining an accurate analysis of the samples is crucial for making a hazardous versus non-hazardous determination. For this reason, prepared sample testing is strongly recommended.

**Important Note:** These types of samples are subject to TCLP procedures for the landfill determination. In general, 20 times the XRF result should be less than the allowable limit for the metal in question. Please contact Innov-X Systems for more details on testing samples versus TCLP regulatory requirements.

## Section 3: Quality Assurance.

Quality assurance is detailed for both the proper use of the analyzer (which is also provided in Method 6200) and for verifying the data quality of in-situ testing. All operators should perform the QC procedure, regardless of their data quality objectives. Method 6200 has strict requirements about quality assurance. Additionally, Innov-X recommends that operators verify the data quality of in-situ test results, if they are using in-situ data to guide their reporting or remediation decisions. Procedures are listed below:

### 3.1: Proper verification of instrument operation

These procedures are taken from EPA Method 6200 and updated to be specific to the Innov-X analyzer. Quality assurance here consists of testing known standards to verify calibration, as well as testing blank standards to determine limits of detection and to check for sample cross-contamination or instrument contamination. EPA Method 6200 provides a detailed procedure, which is provided here in abbreviated form.

Components of instrument QC:

1. An energy calibration check sample at least twice daily
2. An instrument blank for every 20 environmental samples
3. A method blank for every 20 prepared samples
4. A calibration verification check sample for every 20 samples
5. A precision sample at least one per day.
6. A confirmatory sample for every 10 environmental samples

**Energy Calibration Check:** The Innov-X analyzer performs this automatically; this is the purpose of the standardization check when the analyzer is started. The software does not allow the analyzer to be used if the standardization is not completed.

**Instrument Blank:** The operator should use the SiO<sub>2</sub> (silicon dioxide) blank provided with the analyzer. The purpose of this test is to verify there is no contamination on the analyzer window or other component that is “seen” by the x-rays. Method 6200 recommends an instrument blank at least once per day, preferably every 20 samples. For either in-situ or prepared-sample testing, the operator should just test the SiO<sub>2</sub> blank to be sure there are no reported contaminant metals.

**Method Blank:** The purpose of the method blank is to verify that cross-contamination is not introduced into samples during the sample preparation process. Method 6200 recommends following the sample preparation procedures with clean SiO<sub>2</sub> once every 20 prepared samples. This QC step is not required if the operator is not preparing samples.

**Calibration Verification:** Innov-X provides NIST standard reference samples for calibration check by operator. The operator should perform a 2-minute test on a NIST standard. The difference between the XRF result for an element and the value of the standard should be 20% or less. Calibration Verification should be performed upon instrument startup and periodically during testing. Note: Innov-X recommends a calibration check every 4 hours. EPA Method 6200 recommends a calibration check every 20 samples NIST reference standards are generally applicable for Pb, As, Cr, Cu, Zn. Innov-X provides additional reference standards for other RCRA or Priority Pollutant metals including Cd, Se, Ag, Hg, Ag, Ba, Sn, Sb, and Ni.

**Precision Verification:** Quoting from EPA “A minimum of one precision sample should be run per day by conducting from 7 to 10 replicate measurements of the sample. The precision is assessed by calculating a relative standard deviation (RSD) of the replicate measurements for the analyte. The RSD values should be less than 20 percent for most analytes, except chromium, for which the value should be less than 30 percent.

**Confirmatory Sample:** It is recommended that one confirmatory sample is run for every 10 samples collected. According to EPA Method 6200: “Confirmatory samples are collected from the same sample material that is analyzed on site, but are sent to an off-site laboratory for formal analysis. The purpose of a confirmatory sample is to judge the accuracy of the data obtained by analysis on site and to allow corrections, if necessary.”

### **Important Notes about confirmatory samples:**

Innov-X always recommends that customers compare prepared-sample results to laboratory results. To do this, collect and prepare a sample following the protocols of Appendix 1. Take a subsample and submit to the laboratory for analysis. The single largest error in XRF analysis is lack of sample preparation. For the best comparison, always use prepared samples.

### **3.2: Determining data quality of in-situ testing:**

For operators relying extensively on in-situ testing, it is important to determine the data quality of this testing at a given site. *This protocol is not intended for every sample, but rather for a small percentage of samples considered representative of the site.* If the operator can demonstrate that quantitative data is achieved with little or no sample preparation, then the site characterization will be completed much more quickly but correctly.

For example, an operator may be able to demonstrate that the XRF result changes considerably when samples are passed through a 2 mm sieve, but that XRF results do NOT change appreciably upon finer sieving. In this case the operator can conclude that good XRF data is achievable with only 2 mm sieving. Sieving only to this level requires far less time than a more robust sample preparation. A protocol to determine the appropriate level of sample preparation is the following:

1. Delineate a region of soil approximately 4" x 4".
2. Perform several in-situ tests in this area, or collect the top (approximately) quarter inch of soil from this region, bag the soil, test through the bag. In either case, average the results.
3. If you did not bag the in-situ test sample, collect the top (approximately) quarter inch of soil from this region and sieve through the 2 mm sieve provided. Otherwise sieve the bagged sample used for the in-situ test. Thoroughly mix the sieved sample, and place some of the sieved material into an XRF cup, and perform a test of this sample.
4. If the results of this prepared sample differ less than 20% with the average in-situ result, this indicates the soil in this region is reasonably homogeneous. The data quality in this case is probably at the semi-quantitative level, rather than just screening data.
5. If the results differ by more than 20%, this indicates the soil is not very homogeneous, and there are serious particle size effects affecting your in-situ measurements.
6. In this case, sieve the sample through the 250  $\mu$ m sieve. Mix this sample and place a subsample into an XRF cup for testing. If this result differs from the previous by less than 20% then this indicates that at a minimum the 2mm sieving is necessary to achieve higher data quality.

7. If this result differs by more than 20% from the sample sieved through 2 mm, then particle size effects are still affecting the XRF result. In this case samples should be sieved through 125  $\mu\text{m}$  to assure data quality at the quantitative level.

## Section 4: Calibration for Innov-X Portable XRF

The Innov-X analyzer may run three different calibration methods, described below. In nearly all cases, customers use the Compton Normalization method. This method (recognized in EPA 6200) offers speed, ease of use, and generally good accuracy for concentration ranges from the ppm level up to 2-3% concentrations. As most field-testing is seeking to remediate or locate environmental contaminants, the upper limit of the calibration (2-3%) is generally not a limitation. If customers do require a calibration up to 100% concentration (i.e. a pure element) then Innov-X recommends they also include the Fundamental Parameters (FP) software module with the analyzer. The FP module may be added at time of purchase or as an upgrade at any later date.

**Note:** In general customers do not need to calibrate the Innov-X analyzer for soil testing. The analyzer is delivered with a factory calibration, generally based upon the Compton Normalization (CN) method. The CN method has been proven over the past several years to provide a robust calibration generally independent of site-specific soil matrix chemistry. The operator may calibrate the Innov-X system if desired, but calibration is not required to use the analyzer effectively. All customers should follow the QC procedure described in Section 3, which includes a check of the calibration.

The final model is the empirical calibration. In this case, customers run standards to generate calibration curves for various elements in specific soil matrices. Provided the sample is well-prepared, the empirical method generally yields the most accurate result. In our experience, the accuracy gains going from Compton Normalization to Empirical Mode are small and not worth the extra effort in setting up calibration curves. (The greatest source of error for in-field XRF analysis of soil is lack of adequate sample preparation, thus there is little gained in developing a sophisticated empirical calibration if the operator does to grind and homogenize the all measured samples). The empirical calibration module is an optional software package, available for an upgrade fee at the time of purchase, or as an upgrade at any later date.

### Calibration Requirements:

The concentration of an element in a soil sample is well-described by the formula:

$$w_i = \frac{k_i}{M(Z, i)} I_i$$

$k_i$  = calibration constant for element "i"

$w_i$  = concentration of element "i" – the quantity being measured.

$I_i$  = measured x-ray intensity from element "i"

$M(Z, I)$  = Soil matrix value

The factory calibration determines the value of the calibration constants  $k_i$  for each element, and a typical value  $M(Z,I)$ . The calibration method – either CN, fundamental parameters, or empirical – performs the necessary corrections to the value  $M(Z,I)$  that are important for the site-specific soil chemistry. The XRF analyzer uses the measured intensity of each element's fluorescence from the sample, and the calibration data, to produce elemental concentrations.

### **Compton Normalization:**

The Compton Normalization method calibration consists of the analysis of a single, well-characterized standard, such as an SRM or SSCS. The standard data are normalized to the Compton peak. The Compton peak is produced from incoherent backscattering of X-ray radiation from the excitation source and is present in the spectrum of every sample. The matrix affects the way in which source radiation is scattered off the samples. This scatter is directly related to the intensity of the Compton peak. For that reason, normalizing to the Compton peak can reduce problems with matrix effects that vary among samples. Compton normalization is similar to the use of internal standards in analysis for organic analytes.

### **Fundamental Parameters Calibration:**

The fundamental parameters (FP) calibration is a "standardless" calibration. Rather than establishing a unit's calibration curve by measuring its response to standards that contain analytes of known concentrations, FP calibration relies on the known physics of the spectrometer's response to pure elements to set the calibration. Built-in mathematical algorithms are used to adjust the calibration for analysis of soil samples and to compensate for the effects of the soil matrix. The FP calibration is performed by the manufacturer, but the analyst can adjust the calibration curves (slope and y-intercept) on the bases of results of analyses of check samples, such as SRMs which are analyzed in the field.

### **Empirical Calibration:**

The empirical calibration method requires that a number of site-specific calibration standards (SSCS) are used to establish calibration parameters. The instrument response to known analytes is measured and used to create calibration curves. Empirical calibration is effective because the samples used closely match the sample matrix. SSCSs are well-prepared samples collected from the site of interest in which the concentrations of analytes have been determined by inductively coupled plasma (ICP), atomic absorption (AA), or other methods approved by the US Environmental Protection Agency (EPA). The standards should contain all the analytes of interest and interfering analytes. Manufacturers recommend that 10 to 20 calibration samples be used to generate a calibration curve. The empirical method is the least desirable calibration method as it requires that new standards and curves are generated for each site that is analyzed.

## **Section 5: Effects of Moisture on XRF Results:**

Sample moisture has two effects on XRF results:

- ❑ It alters the soil chemistry, since water is another chemical compound that comprises the soil matrix.
- ❑ Moisture impedes the ability to properly prepare samples.

- ❑ Laboratory results are provided on a “dry weight” basis.

### **Effect on Soil Chemistry:**

While the presence of significant moisture does impact the soil chemistry, modern XRF analyzers all perform automatic corrections for variations in soil chemistry from site to site. Indeed, such variations are expected, and that is the reason analyzers use Compton Normalization or fundamental parameters, in order to correct for moisture content changes as well as other differences in soil geochemistry.

EPA Method 6200 states “Moisture content above 20 percent may cause problems, since moisture alters the soil matrix for which the FPXRF has been calibrated.” However, the Compton Normalization or fundamental parameters methods are implemented in order to automatically correct results for changes to the soil matrix. Thus, we believe that soil moisture is not a significant effect on accuracy due to effects of soil matrix, except for the “dilution” effect that can cause discrepancies with laboratory results which is described below.

### **Sample preparation issues:**

The inability to adequately prepare a wet sample is, we believe, the single biggest contributor to errors when testing wet samples. It is very difficult to grind or sieve a wet sample. The highest quality XRF results are generally obtained from prepared samples. If the operator is unwilling to dry the sample to prepare it, comparisons to the laboratory may yield poorer correlation since the samples are not homogeneous.

### **Laboratory Tests on Dry-Weigh Basis:**

Laboratories always dry samples prior to analysis. They report percent weight content based upon a dry sample basis. Portable XRF may often be used to analyze wet samples in the field, and results are thus reported that include the moisture content. Thus, with all other factors the same, the laboratory will report results higher than portable XRF. The results will be higher by the amount of moisture content in the sample. For example laboratory results will be 10% higher compared to XRF results, if the sample contained 10% by weight water when it was tested with XRF. Recall, this applies to samples where other possible sources of error are the same or negligible.

## **Section 6: Comparing XRF Results to Laboratory Results:**

Innov-X strongly recommends that operators compare prepared sample results to laboratory results. This is because prepared-sample results yield the best possible accuracy with portable XRF. Moreover, the most common source of error is due to non-uniform samples. The XRF technique, nor can any analytical technique, properly account for non-uniform sample types.

To perform a comparison between XRF results and laboratory:

1. Collect a sample and prepare it according to the sample preparation guide in Appendix 1.

2. Take a sub-sample (5-10 grams) of the fully-prepared sample, place it into an XRF cup and perform at least a one-minute test on that sample.
3. Send the same sample to the laboratory for wet chemistry analysis.
4. Require the laboratory to use a total-digestion method. If the laboratory does not use a total digestion method, they may not extract all of the elemental metal from the sample. In this case, the lab result will be lower than the XRF result. Incomplete sample digestion is one of the most common sources of laboratory error, thus it is very important to request a total digestion method.

**Example of Error:** The operator collects a bag of sample, performs XRF analysis on one part of the bag, and sends the bag, or part of the bag of sample to a laboratory for analysis. The laboratory reports a very different value than the operator obtained with the XRF.

**Problem:** Since the sample is very non-homogeneous, the operator did not obtain a result that was representative of the entire bag of sample. The lab analyzed a different part of the sample and obtained a very different result due to the non-uniformity of the sample. The solution to this problem is, at a minimum, to test several locations in the bag of sample and report the average value. Also note the differences between the tests, as this is indicative of the non-uniformity of the sample. Operator should send entire bag of sample to the lab, and instruct lab to prepare the sample before removing sub-sample for lab analysis.

**Best Practice:** The operator should homogenize and prepare the entire bag of sample, and then collect a sub sample for XRF testing. After testing, the same sample should be sent to the lab.

## Section 7: Common Interferences:

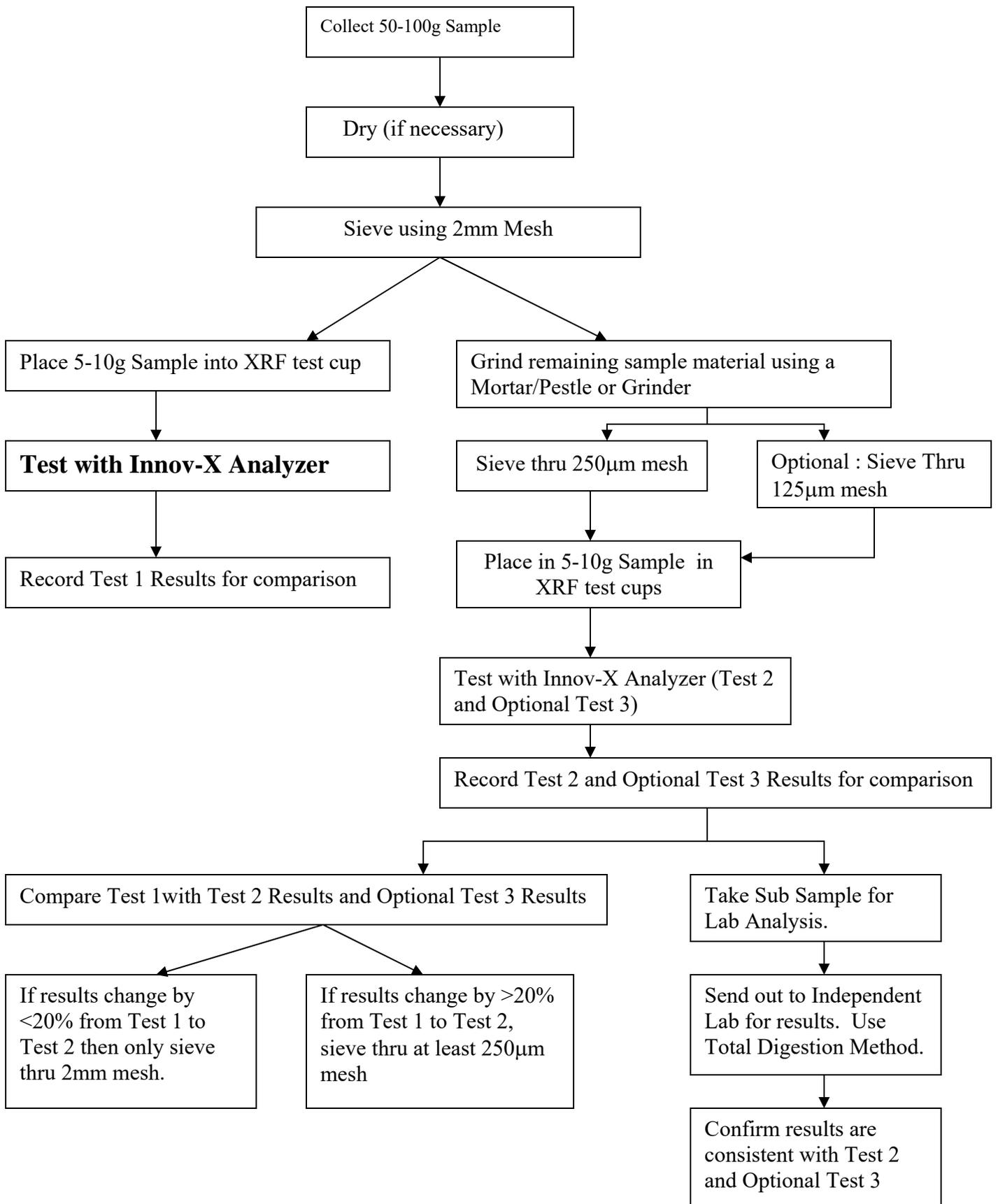
An interference occurs when the spectral peak from one element overlaps either partially or completely with the spectral peak of another. If the XRF is calibrated for both elements (CASE 1) i.e. the one causing the interference and the one being interfered with, it is generally capable of correctly handling the interference. In this case, the element being interfered with may be measured with a poorer detection limit or poorer precision, but the analytical results should still be acceptable for field-portable XRF. If the XRF is not calibrated for the element causing the interference (CASE 2), then the XRF may report the presence of elements not in the sample, or greatly elevated concentrations of elements in or not in the sample.

**Example CASE 1:** Lead and arsenic. Most XRFs are calibrated for lead and arsenic. Lead interferes with arsenic (not vice-versa though). The net effect is a worsened detection limit for arsenic, and poorer precision. The XRF handles the correction automatically, but the precision is affected. The loss of precision is also reported by the XRF. (Please refer to Innov-X Applications Sheet: *In-field Analysis of Lead and Arsenic in Soil Using Portable XRF* for more detail).

**Example CASE 2:** Bromine in the sample, but XRF is not calibrated for bromine. Bromine, as a fire retardant, is being seen more and more in soil and other sample types. For this reason, Innov-X analyzers include Br in the calibration data. If Br is not calibrated, but is present in the sample, the analyzer will report highly elevated levels of Pb, Hg and As. The levels will depend upon the concentration of Br in the sample.

Interferences between elements can be broadly categorized into a) Z, Z-1, Z+1 interferences, and b) K/L interferences. Interference type “a” occurs when high levels of an element of atomic number Z are present. This can cause elevated levels of elements with atomic number Z-1 or Z+1. Generally, portable XRFs have good correction methods, so this interference only causes problems with very high levels of the element in question. Example: High concentrations of Fe (Z=26) in excess of 10% may cause elevated levels of Mn or Co (Z=25 or Z=27 respectively).

The type “b” interference occurs when the L-shell line of one element overlaps with the K-shell spectral line of another element. The most common example is the lead/arsenic interference where the L-alpha line of lead is in nearly the exact same location as the K-alpha line of arsenic.



## Appendix 3: Guide to Product Registration

Generally, the Innov-X portable XRF system must be registered in the state of usage. Registration requirements are somewhat state dependent, but there are many similarities. You may contact Innov-X at 866-4-Innov-X (781-938-5005) to receive specific registration information. Innov-X also maintains sample registrations for every state that we can forward to you.

### Common Registration Features:

Most states require the following for registering an x-ray emitting device that does NOT use radioactive sources:

1. Registration within 30 days of receipt of the analyzer.
2. Annual fee ranging from \$25 to \$100, depending upon the state.
3. Basic registration form with main information described below.

### Common information required on Registration Form, and responses:

Company name, address, phone/fax numbers.

Name of responsible person:	Generally the person designated as the Radiation Safety Officer (RSO).
Name of the manufacturer:	Innov-X Systems, Inc., Woburn, MA
Model of Analyzer:	Alpha XXXX
Tube Operating Parameters:	40 kV, 20 uA current.
Type of Analysis:	Choose Analytical or Industrial (as opposed to radiography, medical, dental, veterinarian, etc.)
Utilization Mode:	Portable or Mobile assuming you will carry system to different locations. Fixed or stationary ONLY if you will always use the analyzer in the docking station

# General Appendix 1

## Technical Specifications

### Description:

Innov-X Systems analyzers are hand-held, battery operated energy dispersive x-ray fluorescence analyzers. They are utilized for the detection and quantification of elements ranging from phosphorus (atomic number 15) through uranium (atomic number 92). Measurable concentrations of elements range from ppm to 100%.

Weight:	2.625 lbs (Base wt.) 3.375 lbs (1.6 kg) with batteries
Excitation Source:	X-ray tube, Ag or W anode, 10-40 kV, 5-50 uA, 5 filter positions
Detector:	Si PiN diode, thermoelectrically cooled, resolution < 280 eV.
Power:	Li-ion batteries, or AC power with Testing Stand
Battery Life:	4-8 hours, depending on duty cycle.
Display:	Color, high-resolution touch screen with variable backlighting on analyzer. Software available for PC/laptop operation also.
Data Storage:	10,000 tests with spectra minimum, expandable to 100,000+ with 1 Gb flash card.
Computer:	HP iPAQ with Intel processor, 64 Mb minimum memory, Windows CE operating system (unless operated from PC).
Optional Accessories:	Bluetooth wireless printing and data transfer, integrated bar-code reader, wireless LAN, other standard PDA accessories.

## Operating Conditions

Temp 0 – 40° C  
Humidity 10 – 90 % RH, no condensation  
Altitude rating 2000 meters

# **Innov-X Analyzer Limited Warranty**

## **General Terms**

EXCEPT AS EXPRESSLY SET FORTH IN THIS LIMITED WARRANTY, INNOV-X SYSTEMS, INC. (INNOV-X) MAKES NO OTHER WARRANTIES OR CONDITIONS, EXPRESSED OR IMPLIED, INCLUDING ANY IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE. INNOV-X EXPRESSLY DISCLAIMS ALL WARRANTIES AND CONDITIONS NOT STATED IN THIS LIMITED WARRANTY. ANY IMPLIED WARRANTIES THAT MAY BE IMPOSED BY LAW ARE LIMITED IN DURATION TO THE LIMITED WARRANTY PERIOD.

This Limited Warranty applies to Innov-X analyzers sold or leased from Innov-X its affiliates, authorized resellers, or country distributors (collectively referred to in this Limited Warranty as (“Innov-X”)).

Innov-X warrants that the analyzer and all its internal components that you have purchased are free from defects in materials or workmanship under normal use during the Limited Warranty Period. The Limited Warranty Period starts on the date of shipment by Innov-X. You may be required to provide proof of purchase or lease as a condition of receiving warranty service. You are entitled to warranty service according to the terms and conditions of this document if a repair to your Innov-X analyzer is required within the Limited Warranty Period.

During the Limited Warranty Period, Innov-X will repair or replace the defective component parts. All component parts removed under this Limited Warranty become the property of Innov-X. In the unlikely event that your Innov-X analyzer has a recurring failure, Innov-X, at its discretion, may elect to provide you with a replacement unit of Innov-X’s choosing that is at least equivalent to your Innov-X analyzer. This is your exclusive remedy for defective products. The repaired or replacement analyzer is warranted for the remainder of the limited Warranty Period.

YOU SHOULD MAKE PERIODIC BACKUP COPIES OF THE DATA STORED ON YOUR ANALYZER AS A PRECAUTION AGAINST POSSIBLE FAILURES, ALTERATION, OR LOSS OF THE DATA. BEFORE RETURNING ANY UNIT FOR SERVICE, BE SURE TO BACK UP DATA AND REMOVE ANY CONFIDENTIAL, PROPRIETARY, OR PERSONAL INFORMATION. INNOV-X IS NOT RESPONSIBLE FOR DAMAGE TO OR LOSS OF ANY PROGRAMS, OR DATA. INNOV-X IS NOT RESPONSIBLE FOR THE RESTORATION OR REINSTALLATION OF ANY PROGRAMS OR DATA OTHER THAN SOFTWARE INSTALLED BY INNOV-X WHEN THE ANALYZER IS MANUFACTURED.

Innov-X does not warrant that the operation of this analyzer will be uninterrupted or error-free. Innov-X is not responsible for damage that occurs as a result of your failure to follow the instructions that came with the Innov-X analyzer.

This Limited Warranty does not apply to expendable parts. This Limited Warranty does not extend to any analyzer from which the serial number has been removed or that has been damaged or rendered defective (A) as a result of accident, misuse, abuse, or other external causes; (b) by operation outside the usage parameters stated in user documentation that shipped with the product; (c) by modification or service by anyone other than (i) Innov-X, or(ii) a Innov-X authorized service provider, (d) installation of software not approved by Innov-X.

These terms and conditions constitute the complete and exclusive warranty agreement between you and Innov-X regarding the Innov-X analyzer you have purchased or leased. These terms and conditions supersede any prior agreements or representations --- including representations made in Innov-X sales literature or advice given to you by Innov-X or any agent or employee of Innov-X --- that may have been made in connection with your purchase or lease of the Innov-X analyzer. No change to the conditions of this Limited Warranty is valid unless it is made in writing and signed by an authorized representative of Innov-X.

### **Limitation of Liability**

IF YOUR INNOV-X ANALYZER FAILS TO WORK AS WARRANTED ABOVE, YOUR SOLE AND EXCLUSIVE REMEDY SHALL BE REPAIR OR REPLACEMENT. INNOV-X'S MAXIMUM LIABILITY UNDER THIS LIMITED WARRANTY IS EXPRESSLY LIMITED TO THE LESSER OF THE PRICE YOU HAVE PAID FOR THE ANALYZER OR THE COST OF REPAIR OR REPLACEMENT OF ANY COMPONENTS THAT MALFUNCTION IN CONDITION OF NORMAL USE.

INNOV-X IS NOT LIABLE FOR ANY DAMAGE CAUSED BY THE PRODUCT OR THE FAILURE OF THE PRODUCT TO PERFORM INCLUDING ANY LOST PROFITS OR SAVINGS OR SPECIAL, INCIDENTAL, OR CONSEQUENTIAL DAMAGES. INNOV-X IS NOT LIABLE FOR ANY CLAIM MADE BY A THIRD PARTY OR MADE BY YOU FOR A THIRD PARTY.

THIS LIMITATION OF LIABILITY APPLIES WHETHER DAMAGE ARE SOUGHT, OR A CLAIM MADE, UNDER THIS LIMITED WARRANTNY OR AS A TORT CLAIM (INCLUDING NEGLIGENCE AND STRICT PRODUCT LIABILITY), A CONTRACT CLAIM, OR ANY OTHER CLAIM. THIS LIMITATION OF LIABILITY CANNOT BE WAIVED OR AMENDED BY ANY PERSON. THIS LIMITATION OF LIABILITY WILL BE EFFECTIVE EVEN IF YOU HAVE ADVISED INNOV-X OR AN AUTHORIZED REPRESENTATIVE OF INNOV-X OF THE POSSIBILITY OF ANY SUCH DAMAGES.

## **Software**

This Limited Warranty does not warrant software products. The Innov-X software installed on your analyzer is covered by the Innov-X Software License.

## **Warranty Period**

The warranty period for a Model XT-245 or Model XT-260 Innov-X analyzer is two years or four thousand hours of use, whichever occurs first. The warranty for all other analyzers is one year or two thousands hours of use whichever occurs first. This warranty does not extend to expendable parts. Extended warranties are available from Innov-X.

## **Warranty Returns**

A Return Material Authorization (RMA) Number must be obtained from the INNOV-X Service Department before any items can be shipped to the factory. Returned goods will not be accepted without an RMA Number. Customer will bear all shipping charges for warranty repairs. All goods returned to the factory for warranty repair should be properly packed to avoid damage and clearly marked with the RMA Number.

## **Warranty Repairs**

Warranty repairs will be done either at the customer's site or at the INNOV-X plant, at our option. All service rendered by INNOV-X will be performed in a professional manner by qualified personnel.

## **Contacting Innov-X**

Be sure to have the following information available before you call Innov-X:

- Analyzer serial number, model name, and model number
- Applicable error messages
- Description of problem
- Detailed questions

## **Methods of Contact**

- Phone: 781-635-5005
- Fax 781-938-0128
- Email [service@Innov-Xsys.com](mailto:service@Innov-Xsys.com)
- Mail & Shipping Address: Innov-X Systems, Inc. 10 Gill Street, Suite Q. Woburn MA 01801

## **APPENDIX B**

### **Laboratory Standard Operating Procedures**



**TestAmerica Denver**

## **Electronic Document Cover**

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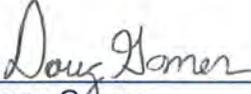
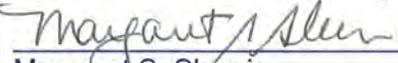
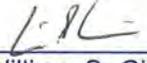
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Electronic Copy Only

**Title: Acid Digestion of Aqueous Samples for Metals Analysis by ICP**

Approvals (Signature/Date):			
	<u>6/26/17</u>		<u>28 June 17</u>
Doug Gomer Technical Specialist	Date	Adam Alban Health & Safety Manager / Coordinator	Date
	<u>6/28/17</u>		<u>6/28/17</u>
Margaret S. Sleeve Quality Assurance Manager	Date	William S. Cicero Laboratory Director	Date

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**1.0 Scope and Application**

**1.1** This standard operating procedure (SOP) describes the acid digestion of aqueous samples by EPA Method 200.7, SW-846 Method 3005A or SW-846 Method 3010A prior to the determination of the concentration of individual metallic elements by inductively coupled plasma atomic emission spectroscopy (ICP). These methods include digestions for total, total recoverable, dissolved, and potentially dissolved analytes (see definitions in Section 3).

**1.2** This SOP is applicable to ground water, surface water, domestic and industrial wastewater, TCLP leachates, and other aqueous media. This SOP is not applicable to oils or other liquids that are not miscible with water.

**NOTE:** Samples that are found to be immiscible with water, e.g., contain oil or other immiscible organic solvents, are subcontracted to other labs that are capable of handling such samples. If during the preparation process it is discovered that the sample is immiscible with water or is biphasic, the analyst notifies the Technical Specialist and Project Manager, who can subcontract the samples to a laboratory with the capability to handle the sample.

**1.3** The following table summarizes the applicability of the various digestion methods referenced in this SOP. All sample digestates are analyzed by ICP in accordance with SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

Method	Title	Summary	SOP Section
3005A/200.7_Prep	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP	Preparation of surface and ground water samples for total recoverable or dissolved metals for analysis by ICP.	10.5
3010A	Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP	Preparation of aqueous samples, EP and mobility procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP.	10.8

**1.4** Sample digestion requirements are established by the laboratory Project Manager before samples are received. TestAmerica LIMS (TALS) method codes are applied to samples at Login to indicate which digestion is to be used for each sample.

**1.5** This procedure can be used for all of the elements listed in Table 1. Additional elements may be analyzed using the digestion methods in this SOP provided the method performance criteria specified in Section 12 and the Quality Control (QC) acceptance criteria specified in Section 9 of this SOP and the ICP determinative SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021 are met.

- 1.6 All samples require digestion prior to analysis, with the possible exception of "direct analysis" of dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples. This must be determined by the laboratory Project Manager before projects start, and is communicated to the analysts through Method Comments in TALS.

## 2.0 Summary of Method

- 2.1 Method 3005A/200.7\_Prep, Total Recoverable, Dissolved Metals or Potentially Dissolved Metals

A representative portion of sample is heated with diluted nitric and hydrochloric acids until substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

- 2.2 Method 3010A Total Metals

A representative portion of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary), and brought up to volume.

## 3.0 Definitions

- 3.1 Dissolved Analyte: The concentration of analyte in an aqueous sample that will pass through a 0.45- $\mu$ m membrane filter prior to acidification (sample is acidified after filtration).
- 3.2 Potentially Dissolved Metals: The concentration of elements in solution after acidifying the sample with nitric acid to pH < 2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45- $\mu$ m membrane filter. This definition is based on the Colorado surface water regulations.
- 3.3 Total Recoverable Analyte: The concentration of analyte determined by analysis of the solution extract of a solid sample or an unfiltered aqueous sample following digestion by refluxing with hot dilute mineral acid(s).
- 3.4 Total Metals: The concentration of elements in an unfiltered sample subject to a more rigorous nitric acid / hydrochloric acid digestion than is used for total recoverable metals.
- 3.5 General Analytical Terms: Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, "Quality Assurance Program," for definitions of general analytical and QA/QC terms.

## 4.0 Interferences

- 4.1 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., talc powdered gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work

areas, and atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

- 4.2 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not miscible with acids. If physical interferences are present, they should be documented in the final report case narrative.
- 4.3 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented in the final report case narrative.
- 4.4 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric acid media.
- 4.5 Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample. Method 3005 or 3010 samples containing more than 1 mg/L silver are redigested at a reduced sample volume and reanalyzed to produce more accurate results. Method 200.7 requires samples to be redigested if the silver is greater than 0.1 mg/L.
- 4.6 Specific analytical interferences are discussed in the ICP determinative methods. See SOPs DV-MT-0012, DV-MT-0019, and DV-MT-0021.

## 5.0 **Safety**

- 5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.
- 5.2 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.
- 5.3 Specific Safety Concerns or Requirements
  - 5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.
  - 5.3.2 Samples that contain high concentrations of carbonates or organic material, or samples that are at elevated pH can react violently when acids are added.

**5.3.3** Care must be taken when handling the digestion tubes. The tubes may become very hot during the digestion procedure. Allow the tubes to cool before attempting to touch the digested samples.

**5.4** Primary Materials Used

**5.4.1** The following is a list of the materials used in this method which have a serious or significant hazard rating.

**5.4.2** 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 SDS for each material before using it for the first time.

<b>Material (1)</b>	<b>Hazards</b>	<b>Exposure Limit(2)</b>	<b>Signs and Symptoms of Exposure</b>
Stock Standard Solutions	Oxidizer Corrosive Poison	5 mg/m <sup>3</sup> as HNO <sub>3</sub>	Toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Nitric Acid (HNO <sub>3</sub> )	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 (HCl)	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.			

## **6.0 EQUIPMENT AND SUPPLIES**

### **6.1 Instrumentation**

- 6.1.1** Digestion blocks, with adjustable heating, capable of maintaining a sample temperature of 90 - 95 °C.
- 6.1.2** Thermometer that covers a temperature range of at least 80 - 110 °C, in increments of 1 °C.
- 6.1.3** Liquid-filled thermometers must have a tag indicating that the accuracy was checked by the QA group within the last 12 months.
- 6.1.4** Digital thermometers must have a tag showing that they were checked within the last three months.
- 6.1.5** See SOP DV-QA-0001 for details of the thermometer calibration procedure.
- 6.1.6** Centrifuge (when the desired method of removing particulates is centrifugation).
- 6.1.7** Calibrated mechanical pipettes with disposable pipette tips. Pipette calibration is checked in accordance with SOP DV-QA-0008.

### **6.2 Supplies**

- 6.2.1** Disposable digestion tubes, with volume accuracy verified to  $\pm 3\%$  gravimetrically prior to use. See SOP DV-QA-0008.
- 6.2.2** Watch glasses, ribbed or equivalent, or disposable digestion tube covers.
- 6.2.3** Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters (No. 6973-2504), for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.
- 6.2.4** Syringes or equivalent filtration apparatus.
- 6.2.5** Re-pipettors or suitable reagent dispensers.
- 6.2.6** Class A volumetric graduated cylinders.
- 6.2.7** pH indicator strips.
- 6.2.8** Plastic digestate storage bottles.

## 7.0 **Standards and Reagents**

- 7.1 Standards must be NIST traceable, where available. Multi-element standards are verified against a second-source standard before they are put into use (the only exception is standards purchased directly from NIST), which is described in SOP DV-QA-0015.
- 7.2 Stock standards are purchased as custom multi-element mixes or as single-element solutions. Standards are logged into the TALS Reagent Module and are assigned unique identification numbers that can be used to access traceability information.
- 7.3 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles.
- 7.4 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.
- 7.5 Standards containing silver must be protected from light using either a cardboard box or amber containers.
- 7.6 Shelf-Life
- 7.6.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, then a one-year expiration will be assigned by the laboratory.
- 7.6.2 Intermediate concentration standards or working standards may be used for up to six months. The expiration date cannot be later than the date assigned to the stock standard.
- 7.6.3 Any suspect standards are re-verified, and replaced if re-verification fails.
- 7.7 Laboratory Control Sample (LCS) Spike Stock Standards

The LCS spike stock standards are custom-made standards purchased from Inorganic Ventures. The standards are designated ICP-SPK-3A (ICP-1) and ICP-SPK-2B (ICP-2) and contain the following elements at ready-to-use concentrations:

**LCS Spike Stock Standards**

<b>Elements in LCS Spike</b>	<b>Concentration in ppm (µg/mL)</b>
Ca, K, Mg, Na	5,000
P, Si	1,000

Elements in LCS Spike	Concentration in ppm (µg/mL)
Al, Ba, Bi, Se, Tl, U, Sn, S	200
Fe, Sr, Li, B, Mo, Ti, As, Th	100
Co, Mn, Ni, Pb, V, Zn, Sb, Zr	50
Cu	25
Cr	20
Cd	10
Ag, Be	5

**7.8 TCLP Spike Stock Standard (TCLP Spike)**

The TCLP spike stock standard is purchased from commercial sources. The stock is a custom-made standard purchased at ready-to-use concentrations and designated as TCLP Spike, as follows:

**TCLP Spike Stock Standard**

Elements in TCLP Spike	Concentration in ppm (µg/mL)
Ba	1,000
Cr, Pb	500
As	300
Cu, Zn	200
Ag, Cd, Se	100

**7.9 TCLP Mercury Spike Solution**

TCLP leachate matrix spike samples are spiked for both ICP elements and mercury at the time of sample preparation but before preservation. The mercury spike standard is prepared by the mercury analyst as the mercury daily spike solution (Hg Daily Spk) at a concentration of 100 µg/L (SOP DV-MT-0015).

**7.10 Reagent Water**

Reagent water must be produced by a Millipore de-ionized system or equivalent and must achieve the performance specifications for ASTM Type II water, i.e., conductivity < 1.0 µmhos/cm; resistivity > 1.0 megohms-cm; silica < 3.0 µg/L. In

addition, the reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs DV-MT-0012, DV-MT-0019, and DV-MT-0021.

7.11 Nitric acid (HNO<sub>3</sub>), concentrated, trace metal grade or better.

7.12 Hydrochloric acid (HCl), concentrated, trace metal grade or better.

## 8.0 Sample Collection, Preservation, Shipment and Storage

Preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time <sup>1</sup>	Reference
Water	HDPE	500 mL	HNO <sub>3</sub> , pH < 2	180 Days	40 CFR Part 136.3

<sup>1</sup> Inclusive of digestion and analysis.

## 9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD/DOE QSM 5.0 or 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

**9.1.4** Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

## **9.1** Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. Ongoing proficiency must be demonstrated by each analyst on an annual basis. See Section 12 for more details on initial demonstrations of capability, analyst training and qualification.

## **9.2** Preparation Batch

A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, a matrix spike (MS), and a matrix spike duplicate (MSD). 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 samples for the MS/MSD pair, then the batch may contain multiple MS/MSD pairs to accommodate client requests. Clients may also request a duplicate LCS (LCSD). In cases where the client has not provided sufficient sample to prepare an MS and MSD, an LCS and LCSD will be prepared instead.

## **9.3** Sample Count

Laboratory-generated QC samples (method blanks, LCSs) are not included in the sample count for determining the size of a preparation batch. The MS and MSD are not included in the sample count unless specifically requested by the client. The prep batch consist of the laboratory generated QC and no more than twenty field samples.

## **9.4** Method Blank (MB)

**9.4.1** 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. When samples are filtered in the laboratory for determination of dissolved metals, then the blank is filtered using a filter of the same type that was used for the samples.

**9.4.2** TCLP method blanks are prepared by taking 10 mL of TCLP leachate fluid (see SOP DV-IP-0012) through the appropriate procedure as described in Section 10. TCLP method blanks are referred to as LB (extraction fluid 1) and LB2 (extraction fluid 2) in TALS and on the final reports.

**9.4.3** One method blank must be processed with each preparation batch. 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. Method blank results are evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

**9.4.4** Acceptance Criteria

The method blank should not contain any analyte of interest at or above  $\frac{1}{2}$  the reporting limit (RL) or at or above 10% of the measured concentration of that analyte in associated samples, whichever is higher. In other words, the sample result must be a minimum of 10 times higher than the blank contamination level. Method blank results that are greater than  $\frac{1}{2}$  the RL may also be reported if the associated sample results fall below the RL and the client accepts the data.

**9.4.5** Corrective Action

If the method blank does not meet the acceptance criteria, the blank and all associated samples in the batch must be re-digested and reanalyzed.

**9.5** Laboratory Control Sample (LCS)

**9.5.1** One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. When samples are filtered in the laboratory for determination of dissolved metals, then the LCS is filtered using a filter of the same type that was used for the samples.

**9.5.2** An LCS for a batch of aqueous samples is prepared by adding 0.5 mL of each of the LCS spike stock standards, ICP-1 and ICP-2 (Section 7.7), to 50 mL of reagent water. This produces the final concentrations shown in Table 1.

**9.5.3** An LCS for a TCLP batch is prepared by adding 0.5 mL of each of the LCS spike stock standards, ICP-1 and ICP-2 (Section 7.7), plus 0.5 mL of the TCLP Spike stock standard (Section 7.8) to 50 mL of the TCLP leachate solution (see SOP DV-IP-0012). This produces the final concentrations shown in Table 2.

**9.5.4** The LCS is used to monitor the accuracy of the analytical process. LCS results are evaluated by the ICP analyst as described in SOP DV-MT-0012. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

**9.5.5** Acceptance Criteria

LCS recovery control limits are set at  $\pm 3$  standard deviations about the historical mean. These limits must not be wider than 85 - 115 % recovery

for Method 200.7 or 80 - 120 % for Method 6010. The control limits are maintained in the LIMS system.

#### 9.5.6 Corrective Action

If the LCS percent recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reprocessed for analysis. One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. This latter case must be documented in an NCM and explained in the case narrative.

### 9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

**9.6.1** A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Normally, one MS/MSD pair is digested with each preparation batch. Samples identified as field blanks, equipment blanks, or rinse blanks are not appropriate for use as the batch MS/MSD.

**9.6.2** Some programs (e.g., South Carolina and North Carolina) require that MS/MSD pairs are run at a 10% frequency. Also, some clients may require unspiked duplicate samples in place of or in addition to an MS/MSD pair. Check special project instructions attached as Method Comments in TALS and any project QASs before starting the batch.

**NOTE:** This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, the LCS and LCSD are used to measure precision.

**9.6.3** If insufficient sample is available to process an MS/MSD pair, then a duplicate LCS must be processed and an NCM generated. The LCS pair is then evaluated according to the MS/MSD criteria. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

**9.6.4** The purpose of analyzing matrix spike samples is to assess the effect of the sample matrix on the accuracy and precision of the analysis. MS/MSD results are evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021. If the MS/MSD results fail to meet control limits while the LCS results are in control, then something about the sample matrix is interfering with the analysis.

**9.6.5** Matrix spikes for aqueous sample batches are prepared by adding 0.5

mL of each of the LCS spike stock standards, ICP-1 and ICP-2 (Section 7.7), to a digestion tube containing 50 mL of the selected sample. The final spike concentrations are shown in Table 1.

- 9.6.6** Matrix spikes for TCLP batches are prepared by adding 0.5 mL of the TCLP Spike stock standard (Section 7.8) plus 0.5 mL of each of the LCS spike stock standards, ICP-1 and ICP-2 (Section 7.7), to 50 mL of the parent TCLP aliquot. A second aliquot is spiked for mercury analysis at by adding 1.5 mL of the 100 mg/L Hg standard (Hg Daily Spk) to 30ml of parent sample. The matrix spike samples are then preserved with HNO<sub>3</sub> to pH < 2. The final spike concentrations are shown in Table 2.

**NOTE:** The MS and MSD must be spiked prior to preservation of the leachate.

- 9.6.7** Acceptance Criteria

The recovery for each analyte must fall within established limits. The relative percent difference (RPD) between the MS and MSD must be less than or equal to the established RPD limit. If any analyte recovery or relative percent difference (RPD) between the MS and MSD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS.

- 9.6.8** Corrective Action

If MS/MSD results fail to meet control limits, but the LCS results are within limits, then samples do not require re-preparation and reanalysis unless the results indicate that a spiking error may have occurred. If the recovery of the LCS also failed acceptance criteria, then corrective action must be taken. Corrective action will include re-preparation and reanalysis of the batch. One possible exception is an LCS recovery for a given element above the upper control limit with no detection for the same element in the samples. This latter case must be documented in an NCM and explained in the case narrative.

- 9.7** Continuing Calibration Verification Standard (CCV)

Continuing calibration verification standards (CCVs) are not digested but are instead created and evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

- 9.8** Second-Source Initial Calibration Verification (ICV) Standard

Initial calibration verification standards (ICVs) are not digested but are instead created and evaluated by the ICP analysts as described in SOPs DV-MT-0012, DV-MT-0019 and DV-MT-0021.

## **10.0** Procedure

- 10.1** One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.
- 10.2** Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.
- 10.3** All data shall be recorded directly on the described forms, logbooks, electronic forms, or directly in TALS at the time of data generation. It is not acceptable to record data on loose papers, scraps of paper, gloves, sample vials, or "Post-It" notes. Data may be recorded on paper bench sheets if the sheets are subsequently scanned and saved in a designated folder on the company server.
- 10.4** All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.
- 10.5** Sample Preparation
- 10.5.1** Samples are typically logged in as either water or solid. Waste such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating sample preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous, but it appears to be a waste (biphasic, oil, sludge-like, organic liquid, lots of sediment, etc.), contact the project manager and the laboratory Technical Specialist for further instructions. It may be necessary to subcontract these samples to a laboratory with the capability to digest organic matrices.
- NOTE:** TestAmerica Denver has not implemented digestion methods for water-immiscible organic matrices, e.g., oils. Samples that are known to be incompatible with TestAmerica Denver digestion techniques are typically subcontracted to other laboratories.
- 10.5.2** All samples are to be electronically checked out of sample control using the TALS Internal Chain of Custody (ICOC) module.
- 10.5.3** Proper sample identification is extremely important in any preparation procedure. Labeling of beakers, digestion tubes, and bottles must be done in a manner to ensure connection with the proper sample.
- 10.5.4** If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab, data review and reporting.

**10.5.5** Guidelines are provided in Appendix 1 on procedures to minimize contamination of samples and standards.

## **10.6** Aqueous Sample Preparation Setup

The following setup procedure must be followed for all aqueous samples prior to performing the specific digestion procedure. The sample preparation procedures for Methods 3005A and 3010A detailed in the following sections are also summarized in work instruction WI-DV-016.

### **10.6.1** Verify sample pH

**10.6.1.1** Measure the sample pH with pH paper using a separate aliquot of sample. This can be done using disposable plastic droppers or pouring the sample on to the pH paper. Do not put the pH paper directly into the bottle. Record the pH on a copy of the internal chain of custody (ICOC). When all of the samples have been tested, initial and date the copy of the ICOC, scan it, and save it to the Metals folder on the G: drive.

**10.6.1.2** All water sample pH's must be verified and documented in the batch record before digestion.

**10.6.1.3** If the pH>2 for a sample requiring acidic preservation, record the job in the Sample Filtration and Preservation Logbook.

**10.6.1.4** If laboratory preservation is required, add 1-2 mL of conc. HNO<sub>3</sub> to the sample. Replace the lid and mix the sample. If the pH is still >2 add another addition of HNO<sub>3</sub>. Do not add more than 5 mL. If the pH is still >2 create an NCM saying the sample will not preserve.

**10.6.1.5** Allow the sample to sit for 24 hours following acidification.

**10.6.1.6** Recheck the pH of the sample. If the pH>2, repeat Section 10.5.1.4 until the pH holds at <2 or 5 mL of HNO<sub>3</sub> has been added. If the pH is still >2 after the addition of 5 mL of HNO<sub>3</sub> create an NCM saying the sample will not preserve.

**10.6.1.7** Samples cannot be digested for 24 hours after preservation. Note the date/time of this pH recheck in the Metals Prep Log in the LIMS.

**10.6.1.8** Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH <2 unless precipitation occurs. Test a small portion of sample to see if precipitation occurs. If a precipitate forms do not acidify the leachate and analyze as soon as possible. Leachates may be digested as soon as they are acidified.

**10.6.2** Select the unfiltered fraction for a total or total recoverable analysis or the

filtered fraction for a dissolved analysis. If requested by the client, select the filtered fraction for a total dissolved analysis. For TCLP and SPLP, select the proper sample leachates.

**10.6.2.1.1** Samples requiring dissolved metals determination are either filtered and preserved in the field or are filtered and preserved by the laboratory as soon as possible after receiving the samples. When filtered in the laboratory, the filtration and preservation are recorded in the Laboratory Sample Filtration and Preservation Logbook, including the preservative type and lot number.

**10.6.2.1.2** Samples and batch QC requiring filtration are to be put into a filtration batch. A filtration batch is to have no more than 20 samples.

**10.6.2.1.3** Filter acceptability is demonstrated by using filters of the same type to filter samples and batch QC samples when preparation batches include samples that were filtered in the laboratory. The results of the analysis of the batch QC samples are used to demonstrate that the filtration process neither adds nor subtracts target analytes from samples. The performance of the filtration process is recorded in TALS.

**10.6.3** Mix the sample by shaking the container.

**10.6.4** Measure and transfer 50 mL of the sample into a digestion tube (record the lot number of the digestion tubes used in the LIMS). When using calibrated digestion tubes, pour the sample into the tube to the 50 mL mark. For TCLP sample batches pour 10 mL of samples and bring to 50 mL with reagent water. Unless specifically required for a project, all samples are measured by volume and not by weight. Record the volume and units on the preparation bench sheet in TALS. If the digestion cup is filled beyond the required mark, the excess sample must not be poured back into the original container, but must be disposed of as waste.

**10.6.5** Mix the sample by shaking the container and then measure two extra aliquots of the sample that is selected for the MS/MSD analysis. Spike each aliquot as described in Section 9.6. Refer to Section 9.6.6 for specific instructions for spiking the selected TCLP sample. Record the standards and pipette identifications in TALS.

**10.6.6** Measure and transfer 50 mL of reagent water into a digestion tube for the method blank. If a determination of dissolved metals is requested (LIMS 3005A), use filtered reagent water for the method blank. For TCLP sample batches, measure 10 mL of the TCLP leachate solution and bring to 50 mL with reagent water for the blank. See Section 9.4 for a detailed description of the method blank.

**10.6.7** Measure and transfer 50 mL of reagent water into a digestion tube for the

LCS and add the spiking solutions as described in Section 9.6.2. For TCLP sample batches, use 10 mL of TCLP leachate fluid and bring to a final volume of 50ml with reagent water for preparing the LCS (Section 9.5.3). Record the standards and pipette identifications in TALS. If determination of dissolved metals is requested and one or more samples were filtered in the laboratory, then filter the LCS using a filter of the same type that was used to filter the sample(s).

**10.6.8** If the analysis is for total recoverable, dissolved metals, or potentially dissolved metals, continue on with Section 10.5. If the analysis is for total metals, skip Section 10.6 and go to Section 10.7.

**10.7** Total Recoverable, Dissolved, or Potentially Dissolved Digestion for Waters by 3005A and 200.7\_Prep.

**10.7.1** Add 1 mL of concentrated HNO<sub>3</sub> and 2.5 mL of concentrated HCl to the sample in the digestion tube.

**10.7.2** Heat at 90-95 °C until the volume is reduced to between 15 and 20 mL. Record the start and stop times, digestion block temperature (observed and corrected) and the thermometer ID in TALS.

**CAUTION:** DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

**10.7.3** Allow the digestion tube to cool in a fume hood.

**10.7.4** Wash down the digestion tube walls with reagent water.

**10.7.5** Add 1.5 mL of concentrated HNO<sub>3</sub> to the digestate.

**10.7.6** Revolume to 50 mL with reagent water. Cap and shake to mix.

**10.7.7** If insoluble materials are present, the sample will be filtered at the instrument by the analyst.

**NOTES:** If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Instead of filtering, the samples may be diluted and mixed and then centrifuged or allowed to settle overnight to remove insoluble material from the supernatant solution.

**10.7.8** The sample is now ready for analysis.

**10.8** Total Metals Digestion for Waters or TCLP Leachates by 3010A

**10.8.1** Add 1.5 mL of concentrated HNO<sub>3</sub> to the sample in the digestion tube.

**10.8.2** Heat at 90-95 °C until volume is reduced to 10 ± 5 mL. Record the start and stop times, digestion block temperature (observed and corrected) and the thermometer ID in TALS.

**CAUTION:** DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

**10.8.3** Allow the digestion tube to cool in a fume hood.

**10.8.4** Add another 1.5 mL portion of concentrated HNO<sub>3</sub> and cover the sample with a watchglass.

**10.8.5** Continue refluxing until the digestion is complete.

**NOTE:** Digestion is complete when the digestate is light in color or does not change in appearance. For most samples the addition of two nitric acid aliquots is sufficient. Additional aliquots of nitric acid may be added if necessary.

**10.8.6** Evaporate to a low volume of 5 to 10 mL. If the sample does go to dryness, the digestion must be started over using a fresh portion of sample.

**10.8.7** Allow the digestion tube to cool in a fume hood.

**10.8.8** Add 2.5 mL of concentrated HCl.

**10.8.9** Cover and reflux for an additional 15 minutes to dissolve any precipitate or residue.

**10.8.10** Wash down the digestion tube walls and watch glass (or digestion tube cover) with reagent water.

**10.8.11** Adjust to 50 mL final volume with reagent water. This must be done volumetrically, and not using a balance.

**10.8.12** If insoluble materials are present, the sample will be filtered at the instrument by the analyst.

**NOTES:** If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Instead of filtering, the samples may be diluted and mixed and then centrifuged or allowed to settle overnight to remove insoluble material from the supernatant solution.

**10.8.13** The sample is now ready for analysis.

## **10.9 Calibration**

**10.9.1** The digestion block temperature must be maintained between 90 and 95 °C. The temperature must be monitored continuously while in use and must be recorded in TALS. The temperature must be monitored by measuring the temperature of reagent water contained in a capped digestion tube that is placed in each digestion block. The thermometer used and the start and end times for all temperature cycles are recorded in TALS.

**10.9.2** The thermometer is calibrated in accordance with SOP DV-QA-0001, Thermometer Calibration Procedures.

## **11.0 Calculations / Data Reduction**

**11.1** This SOP does not produce any analytical data. See the determinative method SOPs DV-MT-0012, DV-MT-0019 or DV-MT-0021 for data analysis and applicable calculations.

### **11.2 Documentation**

**11.2.1** All of the preparation information is recorded and stored in TALS.

**11.2.2** The preparation information includes:

**11.2.2.1** Batch number, job and sample numbers, preparation date, and analyst name;

**11.2.2.2** Matrix and prep type;

**11.2.2.3** Initial sample pH, Initial sample volume and final volume;

**11.2.2.4** Reagent manufacturer and lot number for each reagent used;

**11.2.2.5** Digestion tube lot information;

**11.2.2.6** Standard identification number for each standard used;

**11.2.2.7** Start and stop times for digestions;

**11.2.2.8** Observed and corrected temperature readings during digestion;

**11.2.2.9** Identification numbers of calibrated measuring equipment used (thermometers, balances, pipettes, etc.).

### **11.3 Reporting**

**11.3.1** Reporting units are mg/L for water samples.

**11.3.2** If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor, and the data may require flagging.

**11.3.3** All associated data are entered or uploaded into the LIMS as required.

**NOTE:** Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

**11.4** The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process.

## **12.0 Method Performance**

### **12.1 Method Detection Limit Study (MDL)**

**12.1.1** The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy DV-QA-005P. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD and DOE projects, an MDL verification is performed quarterly.

**12.1.2** The current MDL value is maintained in TALS.

### **12.2 Limit of Quantitation Verification (LOQV)**

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or 5.1. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

### **12.3 Demonstration of Capabilities**

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

**12.3.1** Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

- 12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- 12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.
- 12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

#### 12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

### 13.0 Pollution Control

- 13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.
- 13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

### 14.0 Waste Management

- 14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Plan*.
- 14.2 The following waste streams are produced when this method is carried out:
  - 14.2.1 Expired Chemicals/Reagents/Standards: Contact Waste Coordinator
  - 14.2.2 Acidic waste from sample digests: Waste Stream J.

**NOTE:** Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure

## **15.0 References / Cross-References**

- 15.1** SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.
  - 15.1.1** Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.
  - 15.1.2** Method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.
- 15.2** Method 200.7, Determination of Metals And Trace Elements In Water And Wastes By Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, 1994.

## **16.0 Method Modifications**

### **16.1** Modifications Specific to MCAWW Methods (200.7\_Prep)

It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 10.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness versus an exact volume).

- 16.2** Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above  $\frac{1}{2}$  the reporting limit. Common laboratory contaminants are allowed up to the reporting limit in the blank following consultation with the client.
- 16.3** The referenced methods use 100 mL of sample for digestion. This SOP uses a 50 mL aliquot, with a proportional reduction in digestion reagents. This change is made to allow better control of temperature and potential sample contamination with the use of the digestion block. It is also considered one of the laboratory's hazardous waste reduction initiatives.
- 16.4** The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document states "flexibility to alter digestion volumes is addressed and 'allowed' by the table (3-1)

and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples...” EMSL-Ci has also taken the stance that “reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology.”

## 17.0 Attachments

Table 1. Matrix Spike and Aqueous Laboratory Control Sample Levels

Table 2. TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels

Appendix 1. Contamination Control Guidelines

## 18.0 Revision History

- Revision 10 dated 30 June 2017
  - Annual technical review
  - Updated Section 9.1.2 to include QSM 5.1
  - Added current Section 10.4 referencing support equipment IDs and renumbered remaining sections
  - Added Sections 11.3 and 11.4
  - Updated Section 12.1.1 MDLV language for consistency with other SOPs
  - Added current Section 12.2 regarding LOQVs and renumbered remaining sections
  - Updated the language in Sections 12.3 and 12.4 for consistency with other SOPs
  
- Revision 9 dated 30 June 2016
  - Annual technical review
  - Added “document in the batch record” regarding sample pH to Section 10.5.1.2.
  - Updated Section 9.5.2 to say 10ml of TCLP fluid is added to the method blank.
  - Updated Section 10.5.1.5 to wait 24 hours after acidification before checking pH
  - Converted the note in Section 10.5.2 into subsections 10.5.2.1.1 - 10.5.2.1.3
  - Added definition of filtration batch to section 10.5.2.1.2
  - Updated Section 16.2 to say we control common laboratory contaminants to the reporting limit.
  
- Revision 8 dated 30 June 2015
  - Updated Section 10.5.1.1 to include statement about not putting the pH paper into the bottle
  - Added language to Section 4.5 for clarification
  - Added new Section 10.3 reminding analysts to enter data directly at time of acquisition
  
- Revision 7 dated 31 October 2014
  - Annual technical review
  - Removed reference to SOP DV-IP-0017 for oils in section 1.2
  - Added maximum silver concentration to section 4.5 for method 200.7
  - Updated standard ID’s for sections 7.7 and 7.8 and added Sulfur to the spike list
  - Corrected intermediate standard expirations from three months to six months
  - Removed duplicate analyte spike levels in ICP spike standards
  - Changed references from LIMS to TALS
  - Corrected concentration of Hg Daily spike standard
  - Removed Figures 1 and 2
  - Corrected various grammar and language errors
  - Corrected analyte spike levels in Table 1

- Revision 6 dated 08 October 2013
  - Updated sections 10.4.1.3, 10.4.1.4 and 10.4.1.6 about preservation procedure and removed the comment about recording the amount of acid added in the preservation logbook
  
- Revision 5, dated 15 July 2013
  - Annual review
  - Changed section 10.5.5, 7.3, 9.4, 9.5.2, 9.5.4, 10.3.1, 10.3.2, 10.4, 10.4.4, 10.5.2, 10.6.2, 11.2.2, 12.1.1 and 12.3 to reflect current practices
  - Corrected formatting and grammatical errors
  - Clarified sample matrices for this method in section 1.2
  - Corrected references in table associated with section 1.3
  - Added ICP determinative SOPs to sections 1.5, 4.6, 7.10, 9.5.3, 9.7.4
  - Added 200.7\_Prep whenever 3005A was referenced
  - Edited section 3.5 to reflect current reference
  - Removed note associated with section 5.4.1
  - Added SOP reference to section 6.2.1
  - Removed references to Denver Standards Log and replaces those references with TALS reagent module
  - Correct standard names in section 7.7
  - Removed references to Supplemental Metals Prep Sheet
  - Updated sections 10.4.4, 10.4.6 and 10.4.7 for 10 mL TCLP sample aliquot
  - Added reference to 200.7 in Section 15
  
- Revision 4.7, dated 18 July 2012
  - Annual review
  - Updated Section 9.1, 10.1 and 10.2 to reflect current practice
  - Updated Section 9.7.6 on spiking TCLP aliquots
  - Added section 10.4.1.9 for TCLP preservation
  - Removed Appendix 2. Added reference to work instruction in Section 10.4
  - Updated Figures 1 and 2 to reflect current practice.
  - Formatting and editorial changes throughout
  
- Revision 4.6, dated 24 August 2011
  - Added recommendation to use disposable bulbs for pH checking in section 10.8.1.
  - Added requirement to store samples with a Rush form after preserving in section 10.8.1.2.
  
- Revision 4.5, dated 31 January 2011
  - Change note in section 10.8.1.8 to be 24 hours before preparation.

*Earlier revision histories have been archived and are available upon request.*

**Table 1.**

**Matrix Spike and Aqueous Laboratory Control Sample Levels**

<b>Element</b>	<b>LCS Concentration (ug/L)</b>	<b>Matrix Spike Concentration (ug/L)</b>
Aluminum	2,000	2,000
Antimony	500	500
Arsenic	1,000	1,000
Barium	2,000	2,000
Beryllium	50	50
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	100	100
Calcium	50,000	50,000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1,000	1,000
Lead	500	500
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	500	500
Molybdenum	1,000	1,000
Nickel	500	500
Phosphorous	10,000	10,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	10,000	10,000
Si (as SiO <sub>2</sub> )	21,400	21,400
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Thallium	2,000	2,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	500	500
Zinc	500	500
Zirconium	500	500

**Table 2.**

**TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels**

<b>Element</b>	<b>RL (mg/L)</b>	<b>Regulatory Limit (mg/L)</b>	<b>Spike Level (mg/L)</b>
Arsenic	0.1	5,000	5.0
Barium	1.0	100,000	12.0
Cadmium	0.05	1,000	1.05
Chromium	1.0	5,000	5.2
Lead	0.03	5,000	5.5
Selenium	0.05	1,000	3.0
Silver	0.1	5,000	1.05

## **Appendix 1.**

### **Contamination Control Guidelines**

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.



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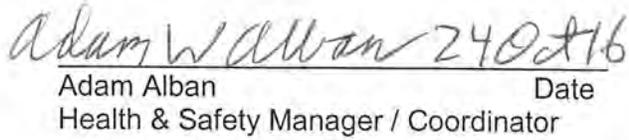
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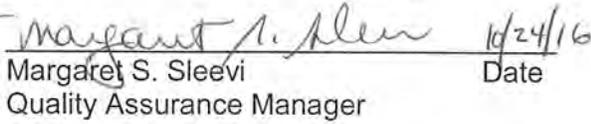
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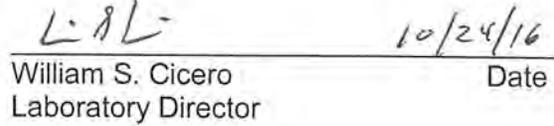
**Title: Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic  
Precipitation Leaching Procedure (SPLP)  
[Method No(s). SW846 1311 and 1312]**

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## 1.0 **Scope and Application**

1.1 This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW-846 Method 1311. The Toxicity Characteristic (TC) of a sample is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a sample. The TC is one of four criteria in 40 CFR Part 261 to determine whether a sample 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 landfill fluids will dominate the acid/base characteristics of the waste. 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 Attachment 1.

**NOTE:** The list in Attachment 1 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 Laboratories Technical Specialists before establishing the instrumental methods for these regulations.

1.3 This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW-846 Method 1312. The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Attachment 1. 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. Currently TestAmerica Denver does not have the capability to digest organic wastes for metals analysis. Therefore if the sample produces a leachate that includes an organic phase, and the client is asking for metals analysis, TestAmerica Denver cannot accept the sample.

1.5 The results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.

1.6 The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits.

- 1.7 If a total analysis of the waste demonstrates that individual analytes are not present in the waste or 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 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.

## 2.0 **Summary of Method**

- 2.1 For liquid samples that contain less than 0.5% dry solid material, the sample, after filtration through 0.6 to 0.8  $\mu\text{m}$  glass fiber filter, is defined as the TCLP leachate and reagent water is used as the blank fluid.
- 2.2 For samples 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 leached with an amount of leach fluid equal to 20 times the weight of the solid phase. For TCLP, the leach fluid employed for the leaching of non-volatile analytes is a function of the alkalinity of the solid phase of the sample. For SPLP, the leach fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals and b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following leaching, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8  $\mu\text{m}$  fiber filter.
- 2.3 If the initial liquid phase of the sample (the filtrate) is miscible with the leachate, then they are combined, prepared, and analyzed together. If not miscible, the filtrate and leachate are analyzed separately and the results can be mathematically combined to yield a volume-weighted average concentration.

## 3.0 **Definitions**

- 3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.
- 3.2 **Leachate**: The TCLP solution generated after solids are tumbled with leaching fluid.
- 3.3 **Filtrate**: The liquid fraction of a sample that passes through a 0.6 to 0.8  $\mu\text{m}$  fiber filter.
- 3.4 **Final Leachate**: The final solution generated from this procedure - either a leachate or a leachate combined with filtrate.

- 3.5** Leach Batch: A Leach Batch as a set of up to 20 field samples of similar matrix that behave similarly and are processed using the same leaching procedure, reagents, and blank fluid type within the same time period. One TCLP leach blank (LB) will be prepared with each TCLP leachate batch.
- 3.6** Percent Wet Solids: The fraction of a sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.
- 4.0** Interferences
- 4.1** Oily samples may present unusual filtration and drying problems. Oils may contaminate the ZHEs and filtration apparatus. Therefore it is important to use filter apparatus designated to oily samples and do extra cleaning after filtration.
- 4.1.1** For oily wastes that filter completely, the filtrate is the leachate and should be sent on for analysis. If the client is requesting metals analysis on these wastes, the sample cannot be analyzed at TestAmerica Denver. For filterable oily wastes requiring semi-volatile organic analysis, the sample should be logged into the LIMS as a waste matrix for method 1311\_T with a waste dilution extraction method 3580. For filterable oily samples requiring volatile organic analysis, the sample should be logged into the LIMS as a waste matrix for method 1311\_Z with a 5030B\_H prep method.
- 4.1.2** For oily wastes that do not filter completely, any filtrate will have to be logged as a separate sample according to Section 4.1.1 above while the portion of the sample that does not filter will have to be leached. The results from the leachate and the filtrate will have to be reported separately and then mathematically re-combined in proportion to give a final result.
- 4.2** 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 leach blanks as described in the Section 9.4 and the individual determinative SOPs.
- 4.3** 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.
- 4.4** 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.
- 4.5** Over exposure of the sample to the environment will result in the loss of volatile components. Samples that are being leached for volatiles should be kept cold. They should not be removed from cold storage until immediately before aliquotting, or alternatively can be kept in an ice bath in the TCLP lab.

4.6 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

## 5.0 **Safety**

5.1 Employees must abide by the policies and procedures in the 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 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2.2 Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized.

CAUTION: Do not open equipment that has been pressurized until it has returned to ambient pressure.

5.2.3 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 over-wraps of the glass jar, may be necessary.

5.2.4 Secure tumbler and extraction apparatus before starting rotary agitation apparatus.

5.2.5 During sample rotation, pressure may build up inside the bottle. Periodic venting of the bottle will relieve pressure. This is more common with samples being leached with TCLP fluid 2. If necessary, secure the lid with duct tape to ensure the vessel stays sealed for the entire leaching period.

5.2.6 Due to the potential for ignition and/or flammability, do not attempt to dry non-aqueous liquid samples in an oven.

5.2.7 Do not attempt to manually stop a rotating piece of equipment. Keep all hanging objects, such as ties, hair, necklaces, etc., away from rotating equipment. Guards must be used when the apparatus is rotating to prevent loose clothing or limbs from getting caught.

5.2.8 Glass vials can break when the caps are being tightened. Cut resistant gloves should be worn whenever caps are being tightened.

**5.2.9** When cleaning ZHE's a methanol rinse is used to remove any residual volatile compounds. After the rinse, the ZHE is put in an oven as a final cleaning procedure. It is very important that after the rinse the ZHE is allowed to dry for two hours in a fume hood before it is put in the oven. If this is not done then methanol vapor will acuminated in the oven resulting in a hazard. This hazard can cause a fire, explosion, or methanol exposure to the face and/or eyes when the door to the oven is opened.

**5.2.10** After performing the procedure, the analyst must separate solid wastes from liquid wastes. This is done by filtering the waste through cloth. The corners and edges of the cloth are gathered together and the liquid is wrung out of the cloth into a drum. The cloth and the trapped solids are then immediately transferred to a waste container. No waste shall be left outside of a closed container.

### 5.3 Primary Materials Used

The following is a list of materials used in this method, which have a serious or significant hazard rating.

**NOTE:** This list does not contain 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 reagent 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 <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid, HNO <sub>3</sub>	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.

Material <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and Symptoms of Exposure
Sodium Hydroxide	Corrosive	2 mg/m <sup>3</sup> (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.
Acetic Acid, Glacial	Corrosive Poison Flammable Liquid and Vapor	10 ppm (TWA)	Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur. Can cause serious damage to skin, including redness, pain, and burns. Contact with eyes may cause severe damage followed by loss of sight.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m <sup>3</sup> (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.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

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

## 6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

**NOTE:** Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

## 6.1 Leach Vessels

**6.1.1** For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Attachment 6). Cleaned by the following steps:

**6.1.1.1** Remove the top and bottom flange from the barrel.

**6.1.1.2** Remove support screens and o-rings from the top flange.

**6.1.1.3** Remove the piston from the barrel and remove the o-rings and wiper seal from the piston.

**6.1.1.4** Wash all parts in hot soapy water, rinse with hot tap water, and rinse with DI water.

**6.1.1.5** Rinse top Flange, barrel, and piston with methanol, and allow to dry in a hood for at least 2 hours before placing in an oven heated to approximately 75°C for at least 4 hours.

**6.1.1.6** O-rings, wiper seals, and screens are placed in a disposable 2L HDPE bottle filled with methanol and tumbled 2-3 hours. They are then allowed to dry in a hood for at least 2 hours before placing in an oven heated to approximately 75°C for at least 4 hours.

**6.1.1.7** Disposable screens can be used instead of re-usable metal screens. Environmental Express part number F2090MM.

**6.1.2** For metals - either disposable borosilicate glass jars (1 gallon, with Teflon lid inserts) or disposable 2 L HDPE (Nalgene® or equivalent) bottles may be used.

**6.1.3** For non-volatile organics – disposable borosilicate glass jars must be used.

**6.2** Vacuum Filtration Apparatus - Capable of 0 - 50 psi. For the filtering of leachates for metal analysis only as the apparatus is constructed of plastics. Cleaned by disassembling completely, washing with warm soapy water, rinsing with hot tap water, rinsing with DI water, and allowing to dry.

**6.3** Stainless Steel Pressure Filtration Apparatus – 142 mm diameter. Capable of 0 - 50 psi. (See Attachment 7). For the filtering of leachates for semi-volatile organics and metals. For the percent wet solids determination. Cleaned by disassembling completely, washing with warm soapy water, rinsing with hot tap water, rinsing with DI water, rinsing with methanol, and allowing to dry.

- 6.4 Acid Washed, Low Metal, Borosilicate Glass Fiber Filters - 0.6 - 0.8  $\mu\text{m}$  (Ahlstrom Grade 26). Certified for low metal content. 14.2 cm in diameter for pressure filter use. 4.7 cm in diameter for vacuum filter use. Glass fiber filters are fragile and should be handled with care.
- 6.5 Glass Fiber Filter Paper – 90 mm in diameter. For use in the ZHE.
- 6.6 Rotary Agitation Apparatus - Multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Attachment 6). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at  $30 \pm 2$  rpm. The RPM is checked before each use.
- 6.7 Gas-Tight Syringes - 100mL capacity, Luer Lock Hamilton 0158330 or equivalent
- 6.8 Top Loading Balance - Capable of 0g – 4000g  $\pm$  0.01g. The balance accuracy is verified each day of use in accordance with SOP DV-QA-0014.
- 6.9 pH Meter and Probe - Capable of reading to the nearest 0.01 unit, and with automatic temperature compensation. Calibrated daily. See Attachment 13 for detailed instructions.
- 6.9.1 Always use fresh aliquots of the pH buffers in fresh cups.
- 6.9.2 Always keep the probe immersed in pH electrode storage solution when not in use.
- 6.9.3 Calibrate the meter using buffers at pH 2, 4, 7, & 10.
- 6.10 Narrow Range pH Strips – Can be used to measure pH in place of the pH meter when dealing with especially oily samples that may damage the pH probe.
- 6.11 Magnetic Stirrer/Hotplate and Stirring Bars – For use in the leach fluid determination.
- 6.12 VOA Vials – 20 mL, with caps and septa. For the storage of leachates for volatile organic compounds analysis.
- 6.13 Glass Jars - 1/2 to 1 gallon, with Teflon lid-inserts. For the storage of leachates for semi-volatile organic compounds analysis.
- 6.14 Nalgene Plastic Bottles – 250mL to 1 L. For the storage of leachates for metals analysis.
- 6.15 Pipette - Calibration checked daily per SOP DV-QA-0008.
- 6.16 Bottle-top Pump – Calibration checked daily per SOP DV-QA-0008 to deliver 96.5mL of water.

**6.17** Log Tag – An automated temperature data recorder used to monitor the temperature of the room during the 16-20 hour leach. See WI-DV-0067 for instructions on how to download the temperature readings.

**6.18** Miscellaneous laboratory glassware and equipment.

**6.19** Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents and Software.xls for the current software to be used for data processing.

**7.0** Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

**7.1** Reagent Water – TestAmerica Denver has three ELGA Analytical water purification systems. The water coming from the ELGA system should be 18-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026. Either water from the ELGA system or bottled HPLC grade water may be used in this procedure.

**7.2** Hydrochloric Acid, 1 N “1N HCl” - For use in leach fluid determination. Add approximately 800mL of reagent water to a 1 liter Class A graduated cylinder. Using a 100mL Class A graduated cylinder, measure out 83mL of concentrated reagent grade HCl and carefully add the acid to the reagent water. Dilute to 1 liter with reagent water. Transfer to a 1 liter glass bottle, cap and shake to mix well.

**7.3** 69%-70% Trace Grade Nitric Acid - For the preservation of final leachates prior to metals analysis. Purchased ready to use.

**7.4** Sodium Hydroxide, 10 N “10N NaOH”- For use in TCLP Fluid #1. Purchased ready to use

**7.5** Glacial Acetic Acid “Acetic Acid”– For use in TCLP Fluid #1 and #2. Concentrated, reagent grade liquid (HOAc).

**7.6** pH Calibration Solutions - Buffered to a pH of 2, 4, 7, and 10. Commercially available. Fresh buffer solution must be used each day of analysis.

**7.7** TCLP Leaching Fluids

The pH of both types of TCLP leaching fluids will be monitored and recorded daily before

use by mixing the fluid well and testing with a calibrated pH meter.

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** TCLP Fluid #1: This reagent is prepared in a manner so that 5.7mL of glacial acetic acid and 64.3mL of 1 N NaOH is diluted to 1 liter in reagent water. When correctly prepared, the pH of this solution is  $4.93 \pm 0.05$ . The laboratory makes this reagent in large quantities by measuring 289mL of 10N NaOH and 256mL of glacial acetic acid and diluting up to 45L of reagent water. (Note that 289mL of 10N base is used instead of 2893mL of 1N base.) The reagent is mixed well as it is being prepared and the pH is checked. If the pH is not within the  $4.93 \pm 0.05$  range, the fluid is not used.
- 7.7.2** TCLP Fluid #2: For every liter of fluid to be prepared, carefully add 5.7 mL glacial acetic acid and dilute up to volume with reagent water. When correctly prepared, the pH of this solution is  $2.88 \pm 0.05$ .
- 7.7.3** For water samples that are determined to be less than 0.5% solids, the leach fluid used to prepare the leach blanks is reagent water.
- 7.8** 60/40 Sulfuric Acid / Nitric Acid - (60/40 weight percent mixture H<sub>2</sub>SO<sub>4</sub>/HNO<sub>3</sub>) For use in SPLP fluids. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.
- 7.9** SPLP Leaching Fluids

SPLP solutions are un-buffered. The pH of SPLP fluids will be checked daily prior to use. Mix well and check with a calibrated pH meter. If not within specifications, the fluid may be discarded and fresh fluid prepared or the fluid must be adjusted using additional acid or reagent water to achieve proper pH.

**NOTE:** All SPLP waste waters **must** be SPLP fluid 1, no matter if they are east or west of the Mississippi.

- 7.9.1** SPLP Fluid #1: This fluid is used for soils from a site that is east of the Mississippi River. Add 60/40 weight percent mixture of sulfuric and nitric acids to approximately 20 liters of reagent water until the pH is  $4.20 \pm 0.05$ . Test with a calibrated pH meter. If the pH is above 4.25 add more acid until the pH is in range. If the pH is below 4.15 dilute by adding more reagent water. Use the spreadsheet described in Attachment 14 to determine how much water to add.
- 7.9.2** SPLP Fluid #2: This fluid is used for soils from a site that is west of the Mississippi River. Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is  $5.00 \pm 0.05$ . Test with a calibrated pH meter. If the pH is above 5.05, add more acid until the pH is in range. If the pH is below 4.95

dilute by adding more reagent water. Use the spreadsheet described in Attachment 14 to determine how much water to add.

**7.9.3** 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. This fluid is also used as the blank fluid for SPLP water samples. If the samples are to be analyzed for common lab contaminants like acetone and methylene chloride by method 8260, the reagent water should first be boiled and purged per DV-MS-0010.

#### **7.10 Metals Spike Standards**

**7.10.1** TCLP Spike – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 2.

**7.10.2** ICP SPK 2B – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 3.

**7.10.3** ICP SPK 3A – Purchased ready to use in 2% nitric acid at the concentrations listed in Attachment 4.

**7.10.4** Hg Daily Spk – Prepared in 1% nitric acid at the concentration listed in Attachment 5.

**7.11** Methanol – Used in cleaning ZHEs and steel pressure filters.

**7.12** Methylene chloride - used to aid in cleaning oil contaminated equipment.

#### **8.0 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. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation.

**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. Water samples should be collected in Teflon lined septum capped vials. Soil samples should be collected in Teflon lined 4 oz jars. Both water and soils should be collected with minimal headspace and stored at  $4 \pm 2$  °C). Samples should be opened only immediately prior to leaching. A second container should be supplied for the percent solids determination.

- 8.4** Samples should be refrigerated to  $4 \pm 2^{\circ}\text{C}$  unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.
- 8.5** The physical state or states of the waste and the analytes of concern determine the minimum TCLP sample collection size. 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.
- 8.5.1** For multi-phasic samples containing between 0.5% and 10% solids, several kilograms of sample are required to complete the analyses.
- 8.5.2** 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.
- 8.5.3** For liquid samples (less than 0.5% 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.
- 8.5.4** If matrix spike or duplicate control samples are requested, additional sample volume is required.
- 8.5.5** If sufficient sample volumes were not received, analyses cannot be started and the project manager should be notified as soon as possible.
- 8.6** 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, then no more acid shall be added and the leachate shall be analyzed as soon as possible.
- 8.7** All leachates for semi-volatile organic analysis should be stored under refrigeration ( $4 \pm 2^{\circ}\text{C}$ ) until analyzed.
- 8.8** Leachates for volatile analysis must be stored under refrigeration ( $4 \pm 2^{\circ}\text{C}$ ) in VOA vials filled to eliminate all headspace.
- 8.9** Samples are subject to appropriate treatment within the following time periods:

PARAMETER	HOLDING TIMES (DAYS)			
	COLLECTION TO START OF LEACH	START OF TCLP TUMBLE TO PREPARATION	START OF TCLP LEACH OR SEMIVOLATILE 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

HOLDING TIMES (DAYS)				
PARAMETER	COLLECTION TO START OF LEACH	START OF TCLP TUMBLE TO PREPARATION	START OF TCLP LEACH OR SEMIVOLATILE PREP EXTRACTION TO ANALYSIS	TOTAL ELAPSED TIME
Other Metals	180	N/A	180	360

**NOTE:** The hold is the same for water and solids.

**NOTE:** The initial holding time is measured from date of collection to date TCLP leach started. (This should be the TCLP leach date in LIMS.) Semi-volatile method prep holding time is measured from the day leach was started 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.

## 9.0 Quality Control

**9.1** The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

**9.1.1** The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Control Program.

**9.1.2** Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, QA/QC Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

**9.1.3** Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

**9.1.4** Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is

described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

- 9.2** Batching Samples - Samples that are less than 0.5% solids (i.e. liquid samples) are batched separately from samples that are greater than 0.5% (i.e. solid samples or multi-phasic samples.)
- 9.3** A Leach Batch is a set of up to 20 field samples of similar general matrix (i.e greater than 0.5% solids or less than 0.5% solids) that behave similarly and are processed using the same leaching procedure, reagents, and blank fluid type within the same time period. One TCLP leach blank (Method Blank) will be prepared with each TCLP leachate batch.
- 9.4** TCLP Leach Blanks - One blank (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch of samples leached that day in a particular vessel type. The leach blanks are generated in the same way as the samples (i.e., blanks will be tumbled and filtered with the samples). Leach fluid is tumbled with the samples in the same type of leach vessel (see Section 6.1) and filtered using the same filtration apparatus (see Section 6.2 and 6.3). Zero Headspace Extraction vessels are uniquely numbered. Each time a new batch is set up the blank should be rotated randomly to a different vessel to ensure all vessels are periodically checked. A vessel cannot be used in the leaching of more than 20 samples before it is used for the leaching of a blank. This is documented in the ZHE spreadsheet.
- 9.5** Laboratory Control Sample (LCS) - A LCS is required with each batch of 20 or fewer samples. The LCS shall be created at the time of the preparative digestion or extraction by spiking an aliquot of the appropriate leach fluid used for that batch. Consult the individual analysis SOPs for additional LCS guidance (i.e., spike amounts, spike levels, recovery criteria, etc.).
- 9.6** 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. An MS/MSD pair is required with each batch. When an MS/MSD pair is not available, an LCS and LCSD are to be used to measure precision.
- NOTE:** Some clients interpret Section 8.2 of SW-846 1311 to mean that a matrix spike must be performed for each specific sample matrix. In other words, if the samples in the batch are visually distinct (clay, soil, sand, wood, plastic, metal) the lab must perform a MS/MSD on each distinct sample matrix type. If the client interprets the method in this way, this will be communicated through the Method Comment "MS per Specific Matrix".
- 9.7** MS/MSD samples will be spiked after final leachate generation at the time of preparative digestion or extraction. Spikes are not to be added prior to the TCLP leaching. For metals, matrix spikes are to be added before preservation with nitric acid.
- 9.8** Consult the individual analysis SOPs for additional guidance on spike compounds and levels.
- 9.9** Consult the individual analysis SOPs for corrective action for blanks, LCSs, and MS/MSDs

## 10.0 Procedure

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 an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must also be documented in an NCM, with a cause and corrective action described.

**NOTE:** The worksheets referred to in this SOP can be found in G:\QA\Edit\FORMS\Organic Prep Forms\TCLP Worksheets Rev 9.

**NOTE:** See Attachment 12 for instructions on how to create batches in the LIMS system "TALS".

### 10.1 WORKSHEET 1, SECTION A, SAMPLE DESCRIPTION – Enter data on Worksheet 1.

**10.1.1** Preliminary TCLP evaluations (percent solids, particle size, selection of leach fluid, and fluid/leachate compatibility) are required to be done using a minimum of a 100 gram aliquot of sample. 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.

**10.1.2** Record the number of phases observed in the sample. 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.

**10.1.3** If the sample has multiple phases and is received in more than one bottle, then the contents of each bottle should be combined in a single larger container prior to processing the sample further. However, the aliquot for volatile analysis should not be combined because that would expose the sample to headspace.

**10.1.4** *LINE A.1* - Record the visible presence of a solid material heavier than water. If the sample contains more than one solid phase (e.g., wood and sediment mixed with water), describe the different phases in an NCM.

**10.1.5** *LINE A.2* - Record the number of liquid phases observed in the sample according to apparent density. 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, a small drop of the liquid can be added to a small amount of water to test the relative density.

**NOTE:** If the sample contains an oil layer, see Section 4.1 for guidance.

**10.1.6** If the sample will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 10.3 (Leach Fluid Determination) for semi-volatile and metals analysis and proceed to Section 10.7 (ZHE Leaching Procedure) for volatile analysis. If only one jar was received, the ZHE procedure (Section 10.7) should be completed before proceeding to Section 10.3 for semi-volatile and metals analysis.

## **10.2** WORKSHEET 1, SECTION B – PERCENT SOLID PHASE

**10.2.1** 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 10.7 (ZHE Extraction Procedure). Otherwise continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.

**10.2.2** Determine Type of Filtration Apparatus Needed –

- If the sample is mostly a non-viscous liquid (water or non-viscous organic liquid) of low solids content (<10%) or a liquid containing highly granular solids, either vacuum filtration or pressure filtration may be used
- If the sample is viscous (sludge or has high solids content), use pressure filtration.
- If the sample is oily, a glass vacuum flask and Büchner funnel can be used to filter the sample.

**10.2.3** *LINE B.1 - Weight of Filter.* Measure and record this value before loading the filter into the filter holder. Assemble the filtration apparatus. Use care when handling the 0.6 to 0.8 µm filter so as not to bend the filter or to contaminate it with trace amounts of oil from your hands.

**10.2.4** *LINE B.3.b – Tare Weight of filtration collection bottle.* Select an appropriate container to collect the filtrate into. Record the weight of the empty container as the tare weight of the filtrate. A plastic bottle can be used if only metals analysis is requested, but a glass container should be used if any organic analyses are requested.

**10.2.5** *LINES B.2.a, B.2.b, and B.2.c - Weight of Subsample for Percent Solids Determination.*

**10.2.5.1** Weigh the full sample container and document this as the gross weight (Line B.2.a). Whenever possible, the entire contents of the sample container should be used in the percent solids determination.

If the entire contents of the sample container are used, then transfer the entire contents to the filtration apparatus. It might be more conducive to filtering if any liquid portion is poured into the filtration apparatus first as not to pre-maturely clog the filter. If necessary, centrifugation can be used as well.

If there is limited sample volume, and the entire contents of the sample container cannot be used in the percent solids determination, then care must be taken to create a representative sub sample by creating a well-mixed slurry before taking the sub-sample.

**10.2.5.2** Weigh the empty sample container with any residual sample and document this as the tare weight (Line B.2.b). The worksheet will then calculate the net weight of the sample used for the percent solids determination in Line B.2.c. If net weight is less than 100 g, an NCM should be written as the percent solids determination should be performed on an aliquot of at least 100 g.

**10.2.6** Slowly apply gentle pressure or vacuum of 10 psi to the filtration apparatus. Allow the sample to filter until no additional liquid has passed through the filter during a 2-minute period.

**10.2.7** Increase 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. This may require many hours to complete. The sample should not be filtered for more than 24 hours to avoid evaporation of the filtrate and thus miscalculation of the percent wet solids. If the sample filtration is not complete in 24 hours, then the client should be contacted.

**NOTE:** Some samples will contain liquid material that does not filter. Do not attempt to filter the sample again by exchanging filters. Viscous liquids or solids that do not pass through the filter are classified as a solid.

**10.2.8** *LINE B.3.a – Gross Weight of Filtrate.* Remove the filtrate collection bottle, weigh and record the gross weight.

**10.2.9** *LINE B.3.c – Net Weight of Filtrate.* The worksheet will calculate the net weight of the filtrate.

**10.2.10** *LINE B.4 – Total Weight of Wet Solids.* The worksheet will calculate the total weight of wet solids by subtracting the net weight of the filtrate (Line B.3.c) from the net weight of the subsample (Line B.2.c)

**10.2.11** *LINE B.5 – Weight Percent of Wet Solids.* The worksheet will calculate the percentage of wet solids in the sample based on weight by dividing the Total Weight of Wet Solids (Line B.4) by the Net Weight of the Subsample (Line B.2.c) and multiplying by 100.

- 10.2.12** *LINE B.3.d – Density of Filtrate.* If the percent solids determination result is greater than 0.5%, then determine the density of the aqueous phase of the filtrate using a calibrated pipette to measure the mass of 1 mL.
- 10.2.13** *LINE B.7* - The worksheet will then calculate the volume of the aqueous phase of the filtrate.
- 10.2.14** *LINE B.8* - If the filtrate is multi-phasic, pour the filtrate into a graduated cylinder. Measure and record the volume of the non-aqueous organic phase. If more than one organic phase is observed, enter “See Below” and provide a description at the bottom of Worksheet 1 and record this in a NCM.
- 10.2.15** Retain the filtrate for use in Section 10.3.3. If the sample is logged for metals analysis only, the filtrate can be stored in a plastic container at room temperature. If the sample is logged for any organic analyses, then the filtrate must be stored refrigerated in a glass container. If the sample is logged for analysis of VOCs and a separate container was not received, then a small portion of this filtrate must be stored refrigerated in a VOA vial with no headspace and an NCM written.
- 10.2.16** If the Weight Percent of Wet Solids in Line B.5 is greater than 5.0%, and semi-volatile and metals analyses are required, proceed to section 10.3. If the Weight Percent of Wet Solids in Line B.5 is greater than 5.0% and volatile analysis is required, proceed to Section 10.7.3.
- 10.2.17** If the Weight Percent of Wet Solids in Line B.5 is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is equivalent to the final leachate. If the sample is logged for method 8260B, refer to Section 10.7.1 (ZHE leaching of 100% Liquid Samples) to generate leachate and blanks for volatile analysis. If the sample is logged for semi-volatiles and metals analysis, generate a leach blank by passing reagent water through a clean filtration apparatus similar to the apparatus used in the percent solids determination of the sample. Deliver the leachates and the associated blank to the appropriate departments along with all completed documentation.
- 10.2.18** If the Weight Percent of Wet Solids in Line B.5 is greater than or equal to 0.5% but less than 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 10.2.19 to complete the percent solids measurement on a dry-weight basis. If it is apparent to the analyst that the sample contains a significant amount of solids (>0.5%), the analyst can proceed to Section 10.2.19 to complete the percent solids measurement on a dry-weight basis to confirm this, or can proceed to Section 10.3 (Particle Size Reduction for Fluid Determination) for semi-volatile and metals analysis and Section 10.7.3 (ZHE Leaching of Samples Less than 100%, but greater than 0.5% Solids)

**NOTE:** If obviously oily (non-aqueous) material is entrained on the filter, do not dry the filter but instead proceed to Section 10.3 (Particle Size Reduction for Fluid Selection). Document in an NCM that the percent wet solids result is most likely biased high due to oily material trapped on the filter and that percent dry solids could not be performed.

### 10.2.19 LINE B.6 – Weight Percent of Dry Solids

NOTE: These steps are required only if it is noticed that a small amount of the filtrate is entrained in the wetting of the filter and the percent wet solids in Line B.5 is  $\geq 0.5\%$  and  $< 5.0\%$ .

- Remove the filter with the wet solids from the filtration apparatus. Take care to remove the entire filter. Often the filter will adhere to the apparatus.
- Dry the filter and solid phase at  $100 \pm 20$  ° C. Record the observed temperature of the oven and the thermometer correction factor in Lines 6.d on Worksheet 1. Allow the filter to dry in the oven for at least 10 minutes.
- Remove the filter from the oven and allow to cool.
- Weigh and record the gross dry weight (Line B.6.a). The Worksheet will calculate the Weight Percent of Dry Solids in Line B.6.c using the equation in Section 11.5. If the Weight Percent of Dry Solids is less than 0.5%, then follow the guidelines in Section 10.2.17 for when percent wet solids is less than 0.5%. If the Weight Percent of Dry Solids is greater than 0.5%, repeat the drying step.
- Weigh and record the second gross dry weight (Line B.6.b). If the two weighings do not agree within 1%, perform additional drying and weighing until successive weights agree within 1%. Record the last two successive weights as Weight 1 and Weight 2 on Lines B.6.1 and B.6.2
- If the Weight Percent of Dry Solids is  $\geq 0.5\%$  and the sample will be extracted for non-volatile constituents, proceed to Section 10.3 (Particle Size Reduction for Fluid Selection) using a fresh wet portion of sample.
- If the Weight Percent of Dry Solids result is  $\geq 0.5\%$  and the sample will be extracted for volatile constituents, proceed to Section 10.7.3 (ZHE Extraction Procedure).
- If the Weight Percent of Dry Solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Follow the guidelines in Section 10.2.17 for when percent wet solids is less than 0.5%.

### 10.3 WORKSHEET 2, SECTION C and D – LEACH FLUID DETERMINATION

If the solid content is greater than or equal to 0.5% and if the sample is being analyzed for metals or non-volatile organic compounds, the type of leaching solution must be determined.

The sub-sample used for fluid selection is taken from the non-filterable solid portion of the sample, but the aliquot must not have been subjected to the oven drying in Section 10.2.19.

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.

For SPLP, refer to Section 7.9 for fluid selection. The client must specify matrix type. Check special instructions, LIMS method, or the PM to determine if the sample is from east or west of the Mississippi River. Document on Line D.3, D.4, or D.5 the fluid type used and then proceed to Section 10.3.3 (Fluid Compatibility)

**NOTE:** All SPLP waste waters **must** be SPLP fluid 1, no matter if they are east or west of the Mississippi.

### **10.3.1** *LINE C.1 – Particle Size Reduction for Fluid Determination*

Reference WI-DV-0058. The sub-sample used for fluid determination must consist of particles less than 1 mm in diameter (versus the less than 1 cm requirement for the material used in the actual leach). The method requires smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full leaching. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

Surface Area Exclusion – Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm<sup>2</sup> per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cuboid shape. If the surface area is less than or equal to 3.1 cm<sup>2</sup> per gram, enter “No” on Line C.1 and prepare an NCM documenting the surface area per gram of sample.

If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size. Enter “Yes” in Line C.1. Document in an NCM how the particle size reduction was performed.

Consult your supervisor and project manager when dealing with unusual sample matrices (e.g., wood, cloth, metal, brick)

### **10.3.2** Determination of Appropriate Leach Fluid

**10.3.2.1** Calibrate the pH meter with fresh aliquots of buffer solution in accordance with the manual. See Section 6.9.

**10.3.2.2** *LINE C.2* – Calibrate a balance per DV-QA-0014 and record the balance ID.

**10.3.2.3** *LINE C.3* - Weigh out a 5.0g ± 0.1g sub sample (less than 1mm particle size) of the solid phase into a 150mL beaker. This sub sample cannot have been subjected to the oven drying in Section 10.2.19

**10.3.2.4** *LINE C.4* – Using a Class A graduated cylinder, or a bottle top pump calibrated per DV-QA-0008, add 96.5mL of reagent water to the sub

sample. Document on Line C.4.a the ID of the pipette or graduated cylinder.

**10.3.2.5** *LINE C.5.a, Line C.5.b*, – Stir vigorously for 5 minutes on a stir plate. Document the time the stirring started on Line C.5.a. Document the time the stirring stopped on Line C.5.b.

**10.3.2.6** *Line C.5.c* - Measure and record the sample pH. If the pH is less than or equal to 5.0, use TCLP Fluid #1. Place an “X” in LINE D.1 and proceed to Section 10.3.3 (Fluid Compatibility) If the sample matrix is especially oily, use narrow-range pH paper to measure the pH instead of the pH meter. This is done to protect the pH probe. Document the use of the narrow-range pH paper in an NCM.

**10.3.2.7** *LINE C.6* - If the pH is greater than 5.0, add 3.5mL of 1 N HCl, using a calibrated pipette. Put a “X” on line C.6 and record the HCl Lot# and the Pipette ID in Lines C.6.a and C.6.b. Mix the sample briefly.

**10.3.2.8** *LINE C.7.a thru C.7.f* – Cover the sample with a watch glass and place the sample in a heated water bath and heat to 50°C to 55°C for 10 minutes. Do not stir the sample during this time. 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. The temperature readings will be taken using a calibrated thermometer that is placed in a random sample in the water bath. Record the observed temperature and the thermometer correction factor.

**10.3.2.9** Measure the pH of the sample. During this pH measurement, do not stir the sample. Document the reading on line 7.f. If the sample matrix is especially oily, use narrow-range pH paper to measure the pH instead of the pH meter. This is done to protect the pH probe. Document the use of the narrow-range pH paper in an NCM.

**10.3.2.10** *LINE D.1 and LINE D.2* – If the pH is less than or equal to 5.0, use Fluid #1 If the pH is greater than 5.0, use Fluid #2.

### **10.3.3** Determination of Filtrate/Leach Fluid Compatibility

Skip this Section if the sample did not yield an initial filtrate from Section 10.2

**10.3.3.1** Place 5mL of the appropriate leaching fluid (determined in the previous step) into a 25mL vial. Add 5mL of the initial filtrate, cap and shake.

**10.3.3.2** If the phases are miscible, the initial filtrate and solid phase leachate will be physically recombined upon completion of the leachate generation. Enter an “X” in LINE D.6 If the phases are not miscible, enter “NO”. The initial filtrate and the solid phase leachate will be prepared and analyzed separately and the results mathematically combined. See Section 11.12.

**10.4 WORKSHEET 3, SECTION E– DETERMINATION OF SAMPLE SIZE FOR BOTTLE LEACH PROCEDURE**

**10.4.1** The aliquot used in the Percent Solids determination described in Section 10.2 may be used for this procedure ONLY if it was not oven dried. If the sample is 100% solid or the preliminary aliquot was not oven dried proceed directly to Section 10.4.2 (Particle Size Reduction for Leaching). If the aliquot from the Preliminary Evaluation was oven dried then, using a fresh aliquot of sample, filter the sample to obtain wet solids and filtrate as described in Sections 10.2.2 through Section 10.2.15. The percent wet solids calculations may need to be repeated in order to correct for sub-sampling error. Then using this new aliquot of wet solids, proceed to Section 10.4.2

**10.4.2** *LINE E.1 – Particle Size Reduction for Leaching*

Reference WI-DV-0058. Evaluate the solid portion of the sample for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the sample 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 to weight ratio is greater than or equal to 3.1 cm<sup>2</sup> per gram. (See Section 10.3.1)

Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick). Scissors or tin snips 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 can be reduced using a jaw crusher. Document in an NCM how unusual samples were handled. Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.

**10.4.3** *LINE E.2* -Calibration check a top-loading balance per DV-QA-0014. Document the Balance ID on Line E.2.

**10.4.4** Determine the total volume of leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to Table 2 below. Note that the volumes listed in Table 2 are the minimum volume required for one extraction and analysis. If possible, extra volume should be prepared for re-extractions and re-analysis. Additional volume for MS/MSD analysis should be provided for at least one sample per leach batch for every requested analysis. When an MS/MSD pair is not available, an LCS and LCSD are required. The samples will be leached at a 20X dilution (i.e. 100g of solids will generate 2000mL of leachate).

**Table 2. Minimum Required Leachate Volume**

<b>Analysis</b>	<b>Required Leachate Volume for TCLP (mL)</b>	<b>Required Leachate Volume for SPLP (mL)</b>
Volatiles	20 (3 x 20mL vials are supplied to provide volume for screening and re-analysis)	40 (3 x 40mL vials are supplied to provide volume for screening and re-analysis)
Semivolatiles	200	1000

Analysis	Required Leachate Volume for TCLP (mL)	Required Leachate Volume for SPLP (mL)
Pesticides	100	1000
Herbicides	100	1000
Metals	100	100

**10.4.5 LINE E.3** - Weigh at least 100g of the solid portion of the sample into an appropriate leach vessel. See Section 6.1 for appropriate leach vessels. Document the weight of the sample to the nearest 0.01g on Line E.3. A minimum sample size of 100g is required. If there is insufficient sample, a NCM is needed. If full suite TCLP is requested, use 150g to generate sufficient leachate.

**10.5 WORKSHEET 3, SECTION F– DETERMINATION OF AMOUNT OF LEACH FLUID FOR BOTTLE LEACH PROCEDURE**

**10.5.1 LINES F.1 through F.4** – *Lot number of Leach fluid.* The worksheet will indicate the correct leach fluid to use as determined in Lines D.1 through D.5. Document the Lot number of the leach fluid used in Lines F.1 through F.4.

**10.5.2 LINE F.5** – *pH of Leach Fluid.* Record the pH of the Leach fluid. Check to make sure the pH of the fluid is still within the specifications in Section 7.7 and Section 7.9. If the pH of the buffered TCLP fluids is not within specifications, ensure the pH meter is properly calibrated and re-check. If the re-check is also not within specifications, discard the fluid and make fresh fluid. If the pH of the un-buffered SPLP fluid is not within specifications, either discard the fluid or adjust the pH by adding more acid or more water. See Section 7.9.

**10.5.3 LINE F.6** – *Volume of Leach Fluid.* The worksheet will calculate the volume of leach fluid to add to each sample based on the weight of the sample in Line E.3 using the formula in Section 11.7. Prepare method blanks by filling similar leach vessels with the same leach fluid used for the samples.

**10.6 WORKSHEET 3, SECTION G– RECORD OF BOTTLE LEACH**

**10.6.1 LINE G.1** – Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and turn on the rotator. The rotator speed must be checked under load every day of use. Count the number of rotations in 15 seconds and multiply by 4 to obtain the rotations per minute (RPM). If the RPM is between 28 and 32, then mark line G.1 “YES”. If the RPM is not between 28 and 32, then tag out the rotator until it can be repaired and move the samples to a rotator that does rotate at the correct speed.

**10.6.2 LINE G.2 and G.3** – Rotate the sample end-over-end for 16-20 hours. Record the leach start date and time on Line G.2. As agitation continues, pressure may build up within the bottle for some types of samples. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented fume hood to relieve any built-up pressure. Due to the higher acidity of TCLP Leach Fluid #2, it is more common for these samples to generate excess pressure. Record the leach stop date and time on Line G.3.

**10.6.3** *LINE G.4 – Temperature of Leach.* The temperature of the room should be  $23 \pm 2^{\circ}\text{C}$ . A data-logging device (LogTag) records the room temperature. After the leach has been stopped, record the thermometer correction factor and the observed minimum temperature and in Line G.4.a and the observed maximum temperature in Line G.4.b. The worksheet will then calculate the actual minimum temp in Line 4.c and the actual maximum temp in Line 4.d.

Download LogTag data according to WI-DV-0067. Click on “Data” tab and print data to PDF. File should be stored with the EXCEL TCLP worksheet files. The LogTag PDF file will then be attached to each corresponding batch in the LIMS system “TALS” per instructions in Attachment 12, Line 13.

If the temperature of the room was not  $23 \pm 2^{\circ}\text{C}$ , the solid fractions of the samples must be re-leached. If there is no volume to re-leach, the client must be contacted. The client must decide if the procedure should be canceled or if the laboratory should continue with a NCM.

**10.6.4** Filter the leachate using vacuum or pressure filtration. This should be done the same day the 16-20 hour leaching was finished. For final filtration of the leachate, the glass fiber filter may be changed, if necessary to facilitate filtration. The entire leachate need not be filtered; however sufficient volume should be filtered to support the required analyses plus extra volume in case of re-extraction, re-digestion and MS/MSD. When an MS/MSD pair is not available, an LCS and LCSD are required. If needed, the leachate can be centrifuged to help facilitate filtration.

**10.6.5** *LINE G.5 – pH of Leachate.* Record the pH of the leachate. If the leachate is especially oily, do not use the pH meter to measure the pH as this may damage the probe. Use narrow-range pH paper instead and write an NCM that the pH was measured using narrow-range pH paper instead of a pH meter.

**10.6.6** If the sample contained no initial filtrate, (i.e the sample was 100% solids) the filtered leachate is defined as the final TCLP leachate. Proceed to Section 10.6.10

**10.6.7** *LINE G.6 Volume of Leachate.* If the sample had an initial filtrate from Section 10.2, then measure the volume of leachate recovered so the leachate and the filtrate can be combined in the correct ratio. If the leachate contains an oil phase, it must be separated and its volume recorded on Line G.6.a. The oil and the filtered leachate must be analyzed separately. If requested, the results can be mathematically re-combined. See Section 11.11 and Section 11.12.

**10.6.8** *LINE G.7 – Volume of initial filtrate for recombination.* The worksheet will use the equation in Section 11.8 to calculate how much of the initial filtrate should be combined with the volume of leachate in Line G.6. Consult Line D.6 to determine if the initial filtrate is compatible to the leachate. If they are compatible, they are to be combined in the correct proportions and mixed well. The combined solution is defined as the TCLP leachate. If the initial filtrate and the leachate are not compatible, they are to be prepared and analyzed separately and the results mathematically combined. See Section 11.11 and Section 11.12. The leachate and the filtrate will have to be logged as separate samples in LIMS.

**10.6.9** *LINE G.8 – Volume of combined initial filtrate and leachate.* The worksheet will calculate the volume of the combined filtrate and leachate using the equation in Section 11.9.

**10.6.10** Leachates for organic analyses should be stored in glass containers at 4°C ± 2°C. Refer to Table 2 to determine how much leachate is needed.

**10.6.11** Leachates for metals analysis should be stored in poly bottles. A 250mL aliquot should be submitted for metals analysis.

**10.6.12** Prepare a MS/MSD sub-sample for metals testing following the steps below. When an MS/MSD pair is not available, an LCS and LCSD are required.

**10.6.12.1** Measure out 50mL of leachate into verified digestion tube. Add 0.5mL of the TCLP Spike described in Section 7.10.1. Add 0.5mL of the Prep Spike 2B described in Section 7.10.2. Add 0.5mL of the Prep Spike 3A described in Section 7.10.3. This 50mL aliquot will be split equally for the MS and the MSD after spiking.

**10.6.12.2** If mercury is requested, measure out an additional 75mL aliquot. Pour two 30mL aliquots from the 75mL aliquot. Add 1.5mL of the mercury spike described in Section 7.10.4 to each of the 30mL aliquots. These two 30mL aliquots will now serve as the MS and the MSD.

**10.6.13** Immediately preserve all leachates for metals by adding 1mL of nitric acid at a time until pH of 2 has been achieved. If after 5mL of acid has been added and the pH is still not 2, do not add more acid, but document final pH in an NCM. If a precipitate starts to form, immediately stop adding acid and document in an NCM.

## **10.7** WORKSHEET 4, ZHE PROCEDURE

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.).

Due to the 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 leachate has been collected. Target compounds will volatilize very rapidly, therefore do not allow the waste, the initial liquid phase, or the leachate to be exposed to the atmosphere any longer than necessary. The sample should be kept cold and not allowed to come to room temperature until it is loaded into the ZHE and all headspace has been purged. Keep the sample in cold storage or in an ice bath.

The ZHE cannot accurately determine percent solids <5%. Go to Section 10.2 if it is apparent that the sample is less than 5% solids. If the sample is apparently greater than 5% solids, but less than 100% solids, go to Section 10.7.3. If the sample is 100% solids, go to Section 10.7.3. If the sample is 100% liquid, proceed to Section 10.7.1

**10.7.1 ZHE Leaching of 100% Liquid Samples.** – This procedure is to be used for samples determined to be 100% liquid per Section 10.2

- 10.7.1.1** Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with reagent water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the sample. At least 80mL of sample should be used. If the piston is 2cm below the top of the cylinder, this will be enough volume for 80mL. By seating the piston as high as possible, you will limit the headspace in the ZHE that will need to be purged later and the potential loss of volatiles.
- 10.7.1.2** Assemble the top flange and run water through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing and will prevent the valve from leaking and reduce the frequency of o-ring replacement.
- 10.7.1.3** *LINE H.2.* - Place the sample in the ZHE body. Place the ZHE body on the ZHE base. Place the top flange on top of the ZHE body and secure tightly. Record the ZHE used on Line H.2
- 10.7.1.4** With the inlet/outlet valve closed, pressurize the ZHE until you hear the piston move upwards.
- 10.7.1.5** *LINE I.6* - Slowly open the inlet/outlet valve to release any headspace. Once liquid appears through the inlet/outlet valve, close the valve and attach a clean gas-tight syringe. Slowly open the valve and collect the filtrate. This filtrate is the final leachate. After all leachate has been collected, remove the syringe from the ZHE and document the filtration completion date and time on Line I.6
- 10.7.1.6** Transfer the leachate from the syringe to 20mL vials for TCLP leachates and 40mL vials for SPLP leachates. Care should be taken not to leave any headspace in the vials. The entire leachate need not be transferred, but three vials should be filled to allow for re-analysis and screening.
- 10.7.1.7** Generate a leach blank using reagent water in the same manner as above. Document in the ZHE spreadsheet which ZHEs were used for samples and which ZHEs were used for method blanks. A ZHE cannot be used for a sample if it has not been used as a method blank in the past 20 uses.

**10.7.2 ZHE Leaching of 100% Solid Samples**

- 10.7.2.1** Consult Worksheet 1 and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor. If the preliminary evaluations indicate the need for particle size reduction, crush, cut, or grind the sample so 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<sup>2</sup> per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cuboid shape. If particle size reduction was necessary, document this on Worksheet 4 as an observation and write an NCM.

To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 °C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.

- 10.7.2.2** Assemble the top flange and run T1 fluid through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing. It will also prevent the valve from leaking and reduce the frequency of o-ring replacement. Place the assembled top flange on top of the body, secure tightly. See Attachment 6.
- 10.7.2.3** Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the required sample. At least 25 grams of sample will be needed. Normally the piston should be seated approximately 2cm below the top of the cylinder, but if the sample is bulky, the piston might have to be seated lower. By seating the piston as high as possible, you will limit the headspace in the ZHE that will need to be purged later and the potential loss of volatiles.
- 10.7.2.4** *LINE H.1* – Calibration check the balance per DV-QA-0014 and record the balance ID.
- 10.7.2.5** *LINE H.2* – Record the ID of the ZHE

**NOTE:** To reduce the time the sample is exposed to the air, the steps described in Section 10.7.2.6 through 10.7.2.9 should be done in quick sequence, working with one sample at a time.

- 10.7.2.6** *LINE H.3* – Place the ZHE cylinder on the balance and tare. Transfer 25 to 25.5g of the sample into the ZHE cylinder. Record the mass on Line H.3. If less than 25 g is used an NCM should be written to document the deviation from the procedure.
- 10.7.2.7** Place the ZHE body on the ZHE base and secure the top flange.
- 10.7.2.8** Close the liquid inlet/outlet valve on the top flange. Pressurize the ZHE until you hear the seals set.

- 10.7.2.9** Slowly open the liquid inlet/outlet valve to release all headspace. Then depressurize the ZHE using the pressure release valve.
- 10.7.2.10** *LINE H.7* – The worksheet will calculate the required volume of leach fluid to add to the ZHE in Line H.7, which is 20 times the mass of the wet solids in the ZHE body (e.g. If 25 g of wet solids were used, then 500 mL of fluid would be required). See the formula in Section 11.7.

Load a clean ZHE that has been specifically designated for blank fluid with the correct volume of TCLP Fluid #1 or SPLP Fluid #3 depending on the analysis requested. Measure the amount of fluid from Line H.7 using a clean 500 mL or 1000 mL graduated cylinder, and pour it into the ZHE with the piston moved all the way to the bottom.

Place the assembled top flange (screens and filter paper aren't necessary) on top of the body holding the blank fluid and secure tightly. Attach the connective tubing to the inlet/outlet valve and with pressure flowing, slowly open the valve, while holding the tubing straight up until all the air has been removed from the line. At the first sign of liquid, immediately close the inlet/outlet valve to prevent the loss of any blank fluid.

Attach the other end of the tubing to the sample ZHE, making sure the pressure relief valve on the bottom of the sample ZHE is left open. Slowly open the inlet/outlet valves on both ZHEs and turn on the pressure, which is attached to the ZHE containing the blank fluid, to allow the fluid to flow into the ZHE containing the sample. When it is determined that all the fluid has been transferred, close the inlet/outlet valve on both ZHEs and remove the transfer line. Observe the valve opening for any leaks. If it is leaking, the valve o-rings will need to be replaced.

Pressurize the sample ZHE and slowly open the liquid inlet/outlet valve to release any air that was introduced into the ZHE with the fluid. Close the valve as soon as liquid appears.

- 10.7.2.11** *LINES H.7a, H.7.b, and H.7.c* – Record the lot number and the pH of the fluid used.
- 10.7.2.12** *LINE I.1.a and LINE I.1.b* – Making sure the pressure relief valve at the bottom is closed, pressurize the ZHE to at least 15 psi. Record the on I.1.a. Let the ZHE sit for at least 15 minutes. Check to make sure the gauge indicates no loss of pressure. Record this check on Line I.1.b. Check the inlet/outlet valve for signs of leakage. If the ZHE shows signs of leakage or the pressure gauge indicates leakage, then the ZHE will be removed from service and repaired. Start the procedure over using either a new ZHE or the repaired ZHE and a fresh aliquot of sample. All repairs and maintenance performed on ZHEs are documented in the ZHE log book. If the ZHE has held pressure and there is no sign of leakage from the inlet/outlet valve then proceed on.

- 10.7.2.13** If the pressure gauge indicates a leak, place the ZHE in a bucket of water and watch for air bubbles. If bubbles are coming from the o-ring at the bottom of the cylinder, clean or replace the o-ring and wipe any contamination from the o-ring grooves. If bubbles are coming from the base pressure relief valve, try seating the valve with your finger or mark the base as having a leaky valve and set aside for repair.
- 10.7.2.14** Generate a leach blank by assembling and loading a ZHE with the same leach fluid used for the samples. Record in the ZHE logbook which ZHEs were used for the leaching of samples and which ZHEs were used for the leaching of blanks. A ZHE cannot be used for the leaching of a sample if it has not been used for the leaching of a blank in the past 20 leaches.
- 10.7.2.15** *LINE I.2 through LINE I.4-* Secure the ZHE in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. Record the start time and the end time on Lines I.3 and I.4. The rotator speed must be checked every day of use under load. Count the number of rotations in 15 seconds and multiply by 4 to obtain the rotations per minute (RPM). If the RPM is between 28 and 32, then mark line I.2 "YES". If the RPM is not between 28 and 32, then tag out the rotator until it can be repaired and move the samples to a rotator that does rotate at the correct speed.
- 10.7.2.16** *LINE I.5.a and LINE I.5.b* – A data-logging device (Log Tag) records the room temperature. The maximum and minimum temperature during the leach is recorded.

If the temperature of the room was not  $23 \pm 2^{\circ}\text{C}$ , the solid fractions of the samples must be re-leached. If there is no volume to re-leach, the client must be contacted. The client must decide if the procedure should be canceled or if the laboratory should continue with a NCM.

Download LogTag data according to WI-DV-0067. Click on "Data" tab and print data to PDF. File should be stored with the EXCEL TCLP worksheet files. The LogTag PDF file will then be attached to each corresponding batch in the LIMS system "TALS" per instructions in Attachment 12, Line 14.

- 10.7.2.17** *LINE I.6* - Remove the ZHE from the rotary agitator and check that the ZHE is still under pressure. Do this by quickly opening and closing the pressure release valve and listening for the release of gas. If the ZHE is not under pressure, then the procedure must be repeated using a fresh aliquot of sample and the ZHE should be taken out of service for maintenance and repair.
- 10.7.2.18** *LINE I.7* – Attach a clean gas-tight syringe to the inlet/outlet valve. The plunger of the syringe should be completely compressed before being attached to the ZHE. Slowly open the inlet/outlet valve and allow the leachate to enter the syringe. If necessary the ZHE can be pressurized

to facilitate the collection of the leachate, but care should be taken not to cause effervescence. After enough leachate has been collected to fill three 20 mL vials (about 75 mLs), remove the syringe from the ZHE. If the sample was multiphasic and the filtrate and leachate are to be recombined prior to analysis, the amount of leachate recovered needs to be entered in Line I.7. This step should be performed the same day the 16 to 20 hour leach is finished.

- 10.7.2.19** *LINE I.7.a* - If the leachate is bi-phasic record the volume of the non-aqueous phase on Line I.7.a. Document in an NCM. The oil phase may need to be analyzed separately and results mathematically recombined.
- 10.7.2.20** Transfer the leachate from the syringe to three 20 mL vials for TCLP leachates or three 40 mL vials for SPLP leachates. Care should be taken not to leave any headspace in the vials. The entire leachate need not be transferred.
- 10.7.2.21** Label all leachates and deliver the leachates and associated blank to the GC/MS Volatiles department along with all completed documentation. The leachates should be stored at  $4 \pm 2$  °C.

### **10.7.3 ZHE Leaching of Samples Less than 100%, but greater than 0.5% Solids**

- 10.7.3.1** Consult Worksheet 1 and examine the sample. If the sample appears to be different from the preliminary information found on the worksheet, consult your supervisor. If the preliminary evaluations indicate the need for particle size reduction, crush, cut, or grind the sample so 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<sup>2</sup> per gram. Weigh the particle, and estimate the surface area based on three dimensions assuming a cuboid shape. If particle size reduction was necessary, document this on Worksheet 4 as an observation.

**NOTE:** To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 °C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.

- 10.7.3.2** Assemble the top flange and run water through the valve and work the o-ring back and forth until it loosens up. This will prevent the o-rings from tearing. It will prevent the valve from leaking and reduce the frequency of o-ring replacement. Place the assembled top flange on top of the body, secure tightly. See Attachment 6.

- 10.7.3.3** *LINE H.4.b* - Weigh 1 to 2 empty gas-tight syringes. Record their combined weight as the tare weight on Line H.4.b. More syringes may be needed if the sample contains a low percent solids value. See Line B.5.
- 10.7.3.4** Place o-rings and wiper seals on the ZHE piston. Moisten the o-rings with water and place the piston in the ZHE body. Adjust the ZHE piston in the ZHE body to the appropriate height in order to contain the required sample. By seating the piston as high as possible, you will limit the headspace in the ZHE that will need to be purged later and the potential loss of volatiles.
- 10.7.3.5** *LINE H.1* – Calibration check the balance per DV-QA-0014 and record the balance ID.
- 10.7.3.6** *LINE H.2* – Record the ID of the ZHE

**NOTE:** To reduce the time the sample is exposed to the air, the steps described in Section 10.7.3.7 through Section 10.7.3.15 should be done in quick sequence, working with one sample at a time.

- 10.7.3.7** *LINE H.3* - Place the ZHE cylinder on the balance and tare. Use the equation in Section 11.10 to estimate how much sample to place into the ZHE in order to ensure 25g of wet solids is included in the aliquot. Transfer the sample into the ZHE cylinder. Record the mass on Line H.3.
- 10.7.3.8** Place the ZHE body on the ZHE base and secure the top flange.
- 10.7.3.9** Close the liquid inlet/outlet valve. Pressurize the ZHE until you hear the seals set.
- 10.7.3.10** Slowly open the liquid inlet/outlet valve to release all headspace. Once liquid starts to come out of the valve, immediately close the valve and attach one of the tared syringes.
- 10.7.3.11** Open the valve again and collect the filtrate. Once the syringe is filled, close the valve and attach an additional tared syringe and repeat until no more filtrate is collected. Increase the pressure of the ZHE 10 psi at a time up to 50 psi until no more filtrate emerges from the ZHE after 2 minutes.
- 10.7.3.12** *LINE H.4.a* - Weigh the full syringes and record their combined weight as the gross weight.
- 10.7.3.13** *LINE H.4.c* – The worksheet will then calculate the net weight of the filtrate using the equation in Section 11.2

- 10.7.3.14** *LINE H.5* – Record the volume of the filtrate by reading the graduations on the syringe(s).
- 10.7.3.15** Transfer the filtrate into vials with no headspace. Label and store the filtrate refrigerated  $4 \pm 2^{\circ}\text{C}$ .
- 10.7.3.16** *LINE H.6* – The worksheet will then calculate the total grams of wet solids remaining in the ZHE using the formula in Section 11.3. If less than 25g of wet solids remains in the ZHE, an NCM should be written to document the deviation from the procedure.
- NOTE:** The ZHE has a maximum capacity of 500mL. Therefore you cannot load more than 25g of solids into the ZHE or else you will not be able to add the appropriate volume of leach fluid.
- 10.7.3.17** *LINE H.8-* The worksheet will then calculate the percent wet solids using the formula in Section 11.4
- 10.7.3.18** Follow steps in Section 10.7.2.10 through 10.7.2.21
- 10.7.3.19** If the initial filtrate from Section 10.7.3.15 is miscible with the leachate (as determined in Section 10.3.3), the leachate and the initial filtrate are directly recombined in the correct proportions.

For samples containing greater than 5% wet solids, the percent wet solids value from the ZHE filtration process should be used to determine the volume of filtrate to re-combine with the leachate. Therefore use the value in Line I.8.b. This approach is required since the percent solids value determined using the pressure filter may differ from the percent solids value determined using the ZHE due to sample variability or differences in the filtration apparatus.

For samples containing less than 5% wet solids, the percent wet solids value from the pressure or vacuum filtration process should be used to determine the volume of the filtrate to re-combine with the leachate. Therefore use the value in Line I.8.a. This approach is required because the ZHE is not appropriate to determine the percent solids of a sample if the percent solids are less than 5%.

Document the volume used in the comments section. For example, if the sample contained less than 5% wet solids and you are using the volume of initial filtrate calculated from Line I.8.a, Note "Sample "ABC" initial filtrate volume calculated from Line I.8.a"

- 10.7.3.20** If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately. If the individual phases are analyzed separately, the results can be mathematically recombined by using the recombination calculation in Section 11.12.

**10.7.3.21** Label all leachates and deliver the leachates and associated blank to the GC/MS Volatiles department along with all completed documentation. The leachates should be stored at  $4 \pm 2$  °C.

## **10.8** Maintenance

- 10.8.1** The pH probe should be replaced when it is noticed that the readings drift or are inconsistent. This should be documented in the pH logbook.
- 10.8.2** ZHE valve o-rings need to be replaced when worn. Scientific Instrument Service part numbers V010 and V012. This should be documented in the ZHE spreadsheet.
- 10.8.3** The ZHE inlet/outlet connector can become damaged and should be replaced with Millipore part number YT3009002. This should be documented in the ZHE spreadsheet.
- 10.8.4** The pressure gauges and pressure release valves on the ZHE base need to be replaced when worn or broken. The pressure release valves can be purchased from Millipore under part number XX6700024.
- 10.8.5** The quick connect on the ZHE base will need to be replaced when worn. Swaglok part number SS-QC4-B-2PM.
- 10.8.6** When working with especially oily samples, disposable ZHE screens are preferred to help prevent cross-contamination.

## **10.9** Troubleshooting

- 10.9.1** When leaching samples with TCLP Fluid #2, applying duct tape to the lids can prevent the samples from leaking.
- 10.9.2** It is advisable to monitor and the temperature of the tumble room throughout the day before samples are set to tumble so that the heaters and air conditioners can be adjusted to keep the temperature in range. If four or more rotators are running at a time, this will generate heat in the room and the door might need to be propped open to keep the room in range. Normally heaters set at 75 °F and air conditioners set at 23°C keep the room in temperature range.
- 10.9.3** When working with a sample that appears to be 100% liquid, do not assume the sample is water miscible. Test the miscibility of the sample in water and methylene chloride.
- 10.9.4** When preparing the leach fluids, it is important to mix the fluids well. This is especially important when making large volumes of fluid.
- 10.9.5** When adjusting the pH of SPLP fluid, do not assume the pH of the reagent water is 7. Test the pH of the water and enter it into the adjustment spreadsheet. Also

if only a very small amount of acid is needed to adjust the pH into range, the acid can be pre-diluted before adding it to the fluid to help in mixing and more accurate measurement.

## 11.0 Calculations and Data Reduction

### 11.1 Weight of Subsample (Line B.2.c)

$$(\text{Net Weight, B.2.c}) = (\text{Gross Weight, B.2.a}) - (\text{Tare Weight, B.2.b})$$

### 11.2 Weight of Filtrate (Line B.3.c) or (Line H.4.c)

$$(\text{Net Weight, B.3.c}) = (\text{Gross Weight, B.3.a}) - (\text{Tare Weight, B.3.b})$$

$$(\text{Net Weight, H.4.c}) = (\text{Gross Weight, H.4.a}) - (\text{Tare Weight, H.4.b})$$

### 11.3 Total Weight of Wet Solids (Line B.4) or (Line H.6)

$$(\text{Wet Solids, B.4}) = (\text{Weight of Subsample, B.2.c}) - (\text{Weight of Filtrate, B.3.c})$$

$$(\text{Wet Solids, H.6}) = (\text{Weight of Subsample, H.3}) - (\text{Weight of Filtrate, H.4.c})$$

### 11.4 Weight Percent Wet Solids (Line B.5) or (Line H.8)

$$(\% \text{ Wet Solids, B.5}) = 100 \times (\text{Wet Solids, B.4}) / (\text{Weight of Subsample, B.2.c})$$

$$(\% \text{ Wet Solids, H.8}) = 100 \times (\text{Wet Solids, H.6}) / (\text{Weight of Subsample, H.3})$$

### 11.5 Weight Percent Dry Solids (Line B.6.c)

$$(\text{Weight percent dry solids, B.6.c}) = 100 \times \frac{(\text{Gross dry weight 2 or 1, B.6.b or B.6.a if B.6.a is blank}) - (\text{Weight of filter, B.1})}{(\text{Weight of subsample, B.2.c})}$$

### 11.6 Volume of Aqueous Filtrate (Line B.7)

$$(\text{Vol. of Filtrate B.7}) = (\text{Weight of Filtrate, B.3.c}) / (\text{Density of Filtrate, B.3.d})$$

### 11.7 Volume of Fluid for Bottle Leach (Line F.6) or ZHE Leach (H.7)

$$(\text{Vol. Fluid, F.6}) = (\text{Weight of Wet Solids, E.3}) \times 20$$

$$(\text{Vol. Fluid, H.7}) = (\text{Weight of Wet Solids, H.6}) \times 20$$

### 11.8 Volume of Initial Filtrate to recombine with Leachate (Line G.7), (Line I.7.a) or (Line I.7.b)

$$(\text{Vol. of Inital Filtrate for Recombination, G.7}) = \frac{(\text{Solids Leachated, E.3})}{(\text{Tot. Wet Solids, B.4})} \times \frac{(\text{Leachate Recovered, G.6})}{(\text{Fluid Added, F.6})} \times (\text{Initial Filtrat, B.7})$$

$$(\text{Vol. of Inital Filtrate for Recombination, I.7.a}) = \frac{(\text{Wet Solids in ZHE, H.6})}{(\text{Tot. Wet Solids, B.4})} \times \frac{(\text{Leachate Recovered, I.6})}{(\text{Fluid Added, H.7})} \times (\text{Initial Filtrat, B.7})$$

$$(\text{Vol. of Initial Filtrate for Recombination, I.7.b}) = \frac{(\text{Weight of Filtrate, H.4.c})}{(\text{Fluid Added, H.7})} \times (\text{Volume of Leachate Recovered, I.6})$$

**11.9 Combined initial filtrate and leachate (Line G.8)**

$$(\text{Combined Filtrate \& Leachate, G.8}) = (\text{Vol of Leachate, G.6}) + (\text{Vol of Filtrate, G.7})$$

**11.10 Weight of Sample to Charge to ZHE**

$$(\text{Weight of Sample}) = 100 \times [20\text{g} / (\% \text{wet solids, B.5})]$$

**11.11 Reporting Conventions for Multi-phase Leachates:**

**11.11.1** 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.

**11.11.2** 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.

**11.11.3** 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."

**11.11.4** Units - regardless of the nature of the sample, all TCLP and SPLP results are reported in units of mg/L.

**11.11.5** For limits and significant figures, consult the appropriate analytical methods

**11.12 Mathematical recombination of analytical results:**

$$\text{Final Analyte Concentration} = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

$V_1$  = 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).

## **12.0 Method Performance**

### **12.1 Demonstration of Capabilities**

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- 12.1.1 Since a spiked aliquot is not appropriate for this procedure, initial and continuing demonstration of capability is documented by collecting data for a completed batch or a method specific test/quiz. An acceptable IDOC is determined by demonstrating that the method required batch QC was performed or the analyst "passed" the test/quiz.
- 12.1.2 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.
- 12.1.3 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.
- 12.1.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

### **12.2 Training Requirements**

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

## **13.0 Pollution Control**

- 13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.
- 13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

## **14.0 Waste Management**

- 14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the

policies in Section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Plan."

**14.2** The following waste streams are produce when this method is carried out:

- 14.2.1** Expired Chemicals/Reagents/Standards – Contact Waste Coordinator
- 14.2.2** Solid waste (post extraction) – Excess Solid Samples - Waste Stream S See Note under Section 5.2.10
- 14.2.3** Aqueous waste (post extraction) - Aqueous Waste from TCLP - Waste Stream T
- 14.2.4** Buffer 4 - Aqueous Waste from TCLP - Waste Stream T
- 14.2.5** Buffers 7 and 10 - Aqueous Waste from TCLP - Waste Stream T
- 14.2.6** Methanol waste - Flammable Solvent - Waste Stream C
- 14.2.7** Methylene chloride waste - Waste Stream B
- 14.2.8** Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

**15.0** References

- 15.1** Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update I.
- 15.2** Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, November 1992, SW-846 Proposed Update II.
- 15.3** Related Documents
  - 15.3.1** Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.
  - 15.3.2** Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.
- 15.4** Technical Background Document and Response to Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.

**16.0** Method Modifications

Item	Method	Modification
1	SW846 1311 & SW846 1312	Section 4.2.1 in both method 1311 and 1312 state "The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure." The laboratory pressurizes the ZHE and waits 15 minutes. This change in timing is supported by the laboratory's requirement to verify that pressure was maintained throughout the extraction process. If there is any loss of pressure at the end of the 16-20 hours the sample is discarded and the process repeated.

Item	Method	Modification
2	SW846 1311	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). This SOP states that samples which have been subjected to the oven drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.
3	SW846 1311	Percent Solids Determination. Section 7.1.2 of the source method indicates that "if the percent wet solids is $\geq 0.5\%$ 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, this SOP states that if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.
4	SW846 1311	Section 7.2.13 of the source method provides no guidance as to how to determine filtrate and leach fluid compatibility. Therefore, this SOP has incorporated a miscibility test into the Preliminary Determinations section.
5	SW846 1311	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.
6	SW846 1311	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 TestAmerica Laboratory Quality Manual 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.
7	SW846 1311	Section 6.4 of the source method states samples "may" be refrigerated unless refrigeration results in irreversible physical change to the waste. This procedures states the samples "should" be refrigerated unless refrigeration results in irreversible physical change to the waste.

## 17.0 Attachments

Attachment 1: Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)

Attachment 2: Metals TCLP Spike

Attachment 3: Metals ICP SPK 2A

Attachment 4: Metals ICP SPK 3A

Attachment 5: Metals Hg Daily Spk

Attachment 6: Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

Attachment 7: Pressure Filtration Device

Attachment 8: TCLP Worksheet No. 1: Sample Description

Attachment 9: TCLP Worksheet No. 2: Selection of Leach Fluid

Attachment 10: TCLP/SPLP Worksheet No. 3: Bottle Leach Procedure

Attachment 11: TCLP Worksheet No. 4: ZHE Leach  
Attachment 12: Instructions for Batching in LIMS  
Attachment 13: Instruction Manual for pH meter.  
Attachment 14: SPLP worksheet for correcting fluid pH.

## 18.0 Revision History

- Revision 10, dated 31, October 2016
  - Removed all references to AFCEE
  - Added current section 3.1 (all following sections re-numbered)
  - Added starting paragraph to section 6.0 to record all equipment IDs
  - Added note to section 6.0 to rotate glassware
  - Clarified RPM testing is to be performed “Before each use” in Section 6.6.
  - Added starting paragraph to section 7.0 regarding reagent grade chemicals
  - Grammatical changes in section 7.7.
  - Added “Note” to Sections 7.9 and 10.3 that all SPLP waste waters must be SPLP fluid 1.
  - Updated section 9.1 and subsections to reflect current practices and consistent verbiage
  - Revised sections 9.4, 10.7.1.7, 10.8.2, 10.8.3 to reflect that a spreadsheet is used for documentation in lieu of the logbook.
  - Added requirement for LCSD when no MS/MSD in sections 9.6, 10.4.45, 10.6.4 and 10.6.12.
  - Revised section 10.2.14 to include that a NCM should be generated to record the observation of a multi-phasic sample.
  - Revised section 10.3.2.8 to reflect the usage of a water bath and not hot plate.
  - Revised section 10.4.2 to state a “surface area to weight ratio” instead of surface area for size reduction.
  - Revised section 10.7.2.10 to reflect ZHEs must be pressurized before slowly opening the inlet and outlet valves.
  - Revised section 10.8.2 to reflect O-ring part No. V011 is not being used. Instead V010 is being employed.
  - Revised section 10.9.2 to reflect the temperature change from that of 30°C to 23°C.
  - Added current section 12.1 regarding IDOCs
  - Updated current section 12.2 to reflect current practices and consistent verbiage
  - Added the current method modification #1 to section 16.
- Revision 9, dated 31, October 2015
  - Added detail to Section 4.5 and 10.7 stating that samples that are to be leached for volatiles should be kept cold until loaded into the ZHE and headspace is purged.
  - Revised Sections 10.6.1 and 10.7.2.15 to state that the rotation speed of the rotators must be checked under load each day of operation.
  - Revised Sections 10.6.4 and 10.7.2.18 to state that the leachate should be filtered the same day the 16-20 hour leach is completed.
  - Revised wording in Section 10.4.5 to match Section 10.4.2.
- Revision 8, dated 31, May 2015
  - Reformatted SOP.
  - Added comment to Section 1.4 to state that TestAmerica Denver cannot digest organic waste for metals analysis.

- Section 4 was revised to discuss how organic waste samples will be treated. In the past organic liquids were assumed to be 100% liquid. The SOP was revised to state that organic liquids will be filtered to determine the percent solids.
- Section 5.2.5 was revised to add details to the types of samples more likely to cause pressure to build up in the leach vessel.
- Section 6 was revised to include the LogTag temperature recording device.
- Section 9.2 was revised to clarify samples that are multi-phasic or solid per the procedure will be batched separately from samples that are liquid per the procedure.
- Sections 3.4, 9.3 and 9.4 were revised to state that one leach blank will be prepared instead of “a minimum of one”.
- Section 10.3 was revised to clarify that the steps to determine the leach fluid type are performed on the solid fraction of the sample.
- Section 10.5.2 was revised to instruct the analyst on corrective action if the pH of the leach fluid is not within specifications.
- Section 10.6.3 and Section 10.7.2.16 were revised to instruct the analyst on corrective action if the temperature of the room during the leach is outside of the control limits.
- Sections 10.4.2 and 10.3.1 were revised to reference WI-DV-0058.
- Revision 7, dated 31, May 2014
  - Revised Section 6.9 to call for the pH meter to be calibrated at pH 2, pH 4, pH 7, and pH 10. Revised Attachment 13.
  - Added narrow range pH strips to Section 6 and revised Section 10 to allow their use as an alternative to the pH meter for oily samples.
  - Added the option to use disposable ZHE screens to Section 6.
  - The instructions in Section 7.7 on how to prepare TCLP fluids were revised to more closely match the source method.
  - Updated Section 7.10.2 for spike used to 2B
  - Attachment 14 was added to aid in the preparation of SPLP fluids. Section 7.9 was revised to instruct the analysts to use the spreadsheets when adjusting the pH of the SPLP fluid.
  - Section 9.1.2 was revised to state that this procedure meets all criteria for DoD QSM 5.0 unless otherwise stated.
  - Section 9.3 was revised to clarify how samples are batched by matrix. A note was added to Section 9.6 to clarify the required frequency of MS/MSD samples.
  - Revised Section 10.3.2 to remove the accuracy criteria for the bottle-top pump. Instead a reference was made to the SOP DV-QA-0008 which dictates the requirements for accuracy and precision of bottle-top pumps.
  - Revised Section 10.3.2 to instruct the analyst to cover the sample with a watch glass and to not stir the sample during the 10 minute heating process or during the pH measurement after the 10 minute heating process. This was done to more closely match the source method.
  - Revised Section 10.6.3.1 to instruct the analyst to attach the “Chart” data page from the temperature recording device (LogTag) instead of the “Summary” data page.
  - Added a comment to Section 10.6.10 to direct the analyst to Table 2 to ensure enough leachate is delivered.
  - Updated Section 10.6.12 for Spike used to 2B
  - Added Troubleshooting and Maintenance sections to Section 10.
  - Updated Attachment 3 to include Sulfur and changed name to 2B

- Revised Attachment 6 to show the Cylinder o-ring and the wiper seal on the ZHE.
- Revised Attachment 7 to show the o-ring on the Pressure Filter.
- Revision 6, dated 31, May 2013
  - Section 5.2.10 was added and Section 14.2 were revised to address safety issues with waste handling.
  - Section 6.9 was revised and Attachment 13 was added because the lab acquired a new pH meter.
  - Section 8.9 was revised to show holding times are calculated from the beginning of the leaching procedure.
  - Section 9.1 and 10.0 were revised to reflect current practice.
  - Sections 10.2.19, 10.3.2, 10.6.3 and all Worksheets were revised to instruct the analysts to record the actual and observed temperatures and the thermometer correction factors.
  - Worksheet #3 was revised to indicate the correct leach fluid to use as determined in Lines D.1 through D.5. Section 10.5.1 was revised to reflect this change.
  - Section 10.7 was revised to instruct the analyst to be especially cautious to minimize the samples' exposure to the atmosphere as much as possible to reduce the loss of volatiles.
  - Section 10.7.1.2, Section 10.7.2.3, and Section 10.7.3.4 were revised to instruct the analyst to seat the piston in the ZHE as high as possible when loading to limit the sample's exposure to the atmosphere.
  - A note was added to Section 10.7.2.5 and Section 10.7.3.6 to instruct the analyst to work with one sample at a time to limit the samples' exposure to the atmosphere.
  - Section 10.7.2.6 and Section 10.7.3.7 were revised to instruct the analyst to aliquot the sample directly into the ZHE instead of first aliquotting it into a weigh-boat and transferring it to the ZHE. This was done to limit the samples' exposure to the atmosphere and is now possible because the laboratory utilizes a 3kg mass in the daily balance calibration.
- Revision 5, dated 11, May 2012
  - This procedure was revised to require the use of a 25g aliquot in the zero headspace extractor.
  - This procedure was revised to instruct analyst to measure the mass of sample used in the ZHE procedure on a balance using a weigh boat instead of taring the ZHE body on the balance. This was done because the total mass of the ZHE body and sample exceeds all standard masses available for daily balance calibration checks.
  - This procedure was revised to more accurately reflect how the laboratory is preparing the TCLP Fluid #1 in large quantities.
  - This procedure was revised to remove the requirement that blank fluid be prepared using nitrogen-purged water when volatiles are requested. Water from the laboratory's ELGA purification systems were tested for volatiles, and no volatiles were detected, therefore water from the ELGA systems will be used for the preparation of all leach fluids.
  - Revised section 10.6.12 to properly reflect current practices regarding the MS/MSD spiking procedure for metals analysis.
- Revision 4, dated 25 May, 2011

- Revised Section 4.1 to change instructions on how oily samples should be logged in LIMS.
- Added detail to Section 6.1.1 on how to clean the ZHE apparatus.
- Added a bottle-top pump to the equipment list in Section 6.
- Revised Section 7.1 to state that the water from the ELGA purification system should be 18 to 18.2 Mohm-cm.
- Added additional clarification to Section 7.7 and Section 7.9 on how the TCLP blank fluids are prepared with nitrogen purged water when volatile analyses are requested.
- Section 9.2 was revised to remove the requirement that samples have to be batched separately if the bulk matrix is visibly different. Samples are only batched separately based on % solids determination or per client request.
- Added more detail to Section 10.2.5 on aliquoting samples for percent wet solids determination.
- Revised Section 10.3.2 to require the documentation of exactly how much water was added to the sample, exactly what time the sample was placed on the stir plate, exactly what time the sample was removed from the stir-plate, exactly what temperature the sample was on the hot plate and the exact times the sample was on the hot plate.
- Added Section 10.6.3.1 and Section 10.7.2.16.1 to describe LogTag download and file retention procedure.
- Revised Section 10.6.11 and 10.6.12 to make changes to how the leachates are spiked and preserved for metals analysis.
- Revised Section 10.7.1.3 to state “Nitrogen-purged water” as opposed to “DI water”.
- Revised Section 10.7.2.7 to state “T1 fluid prepared with nitrogen-purged water” as opposed to “DI water”.
- Revised Section 10.7.2.18 to remove duplicated verbiage.
- Added Section 10.7.3.19.3 to instruct the need to document which calculated volume of initial filtrate was combined with the ZHE leachate.
- Updated Attachments 8 thru 11 to include the revisions made above.

*Earlier revision histories have been archived and are available upon request.*

**Attachment 1.**  
**Toxicity Characteristic Analytes and Regulatory Levels (Final Rule)**

<b>Contaminant</b>	<b>mg/L</b>
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresols	200.0
m-Cresols	200.0
p-Cresols	200.0
Total Cresols (used if isomers not resolved)	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor (& epoxide)	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

**Attachment 2**  
**Metals TCLP Spike**

<b>Component</b>	<b>Concentration (ug/mL)</b>
Silver	100
Arsenic	300
Barium	1000
Cadmium	100
Chromium	500
Copper	200
Lead	500
Selenium	100
Zinc	200

**Attachment 3.**  
**Metals ICP SPK 2B**

<b>Component</b>	<b>Concentration (ug/mL)</b>
Boron	100
Molybdenum	100
Antimony	50
Silicon	1000
Tin	200
Titanium	100
Zirconium	50
Sulfur	20

**Attachment 4.  
Metals ICP SPK 3A**

<b>Component</b>	<b>Concentration (ug/mL)</b>
Silver	5
Aluminum	200
Arsenic	100
Barium	200
Beryllium	5
Calcium	5000
Cadmium	10
Cobalt	50
Chromium	20
Copper	25
Iron	100
Potassium	5000
Lithium	100
Magnesium	5000
Manganese	50
Sodium	5000
Nickel	50
Phosphorus	1000
Lead	50
Selenium	200
Strontium	100
Thorium	100
Thallium	200
Uranium	200
Vanadium	50
Zinc	50
Bismuth	200

**Attachment 5.**

**Metals Hg Daily Spk**

<b>Component</b>	<b>Concentration (mg/L)</b>
Mercury	0.1

### Attachment 6. Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

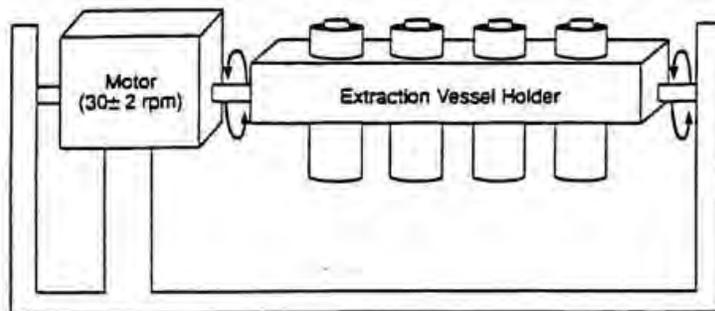
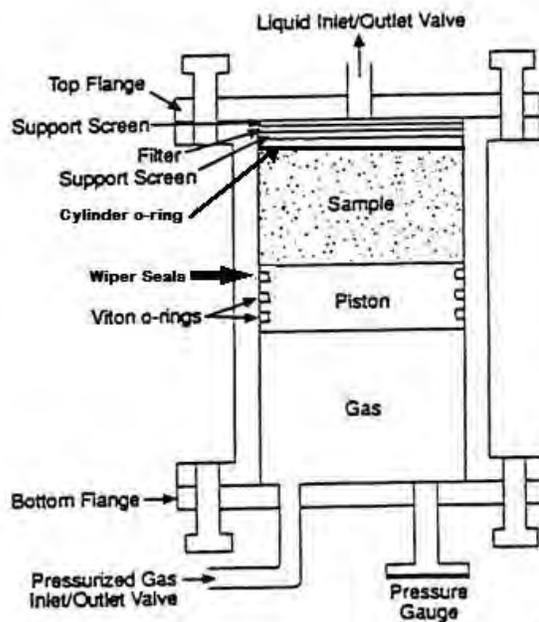
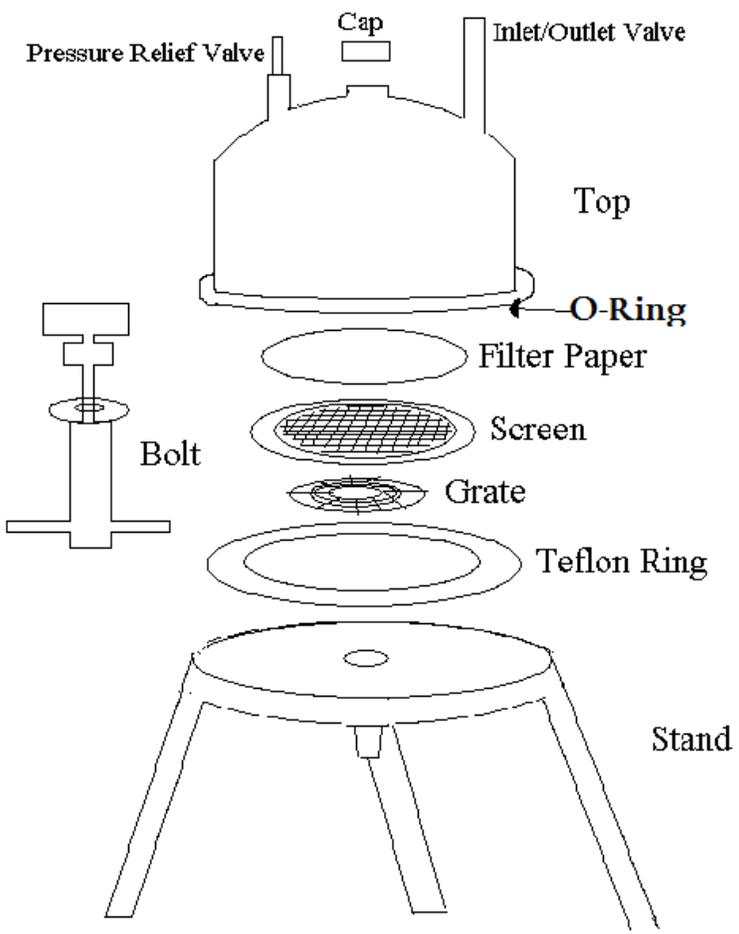


Figure 1. Rotary Agitation Apparatus



**Attachment 7  
Pressure Filtration Device**



**Attachment 8  
 Worksheet No. 1  
 TCLP**

Analyst:	DV-IP-0012									
Date:	TCLP/SPLP Worksheet No. 1									
	Sample Description									
Login No.										
Sample No.										
<b>A. Sample Description</b>										
Number of phases										
1. Solid										
2. Liquid										
a. lighter than water										
b. water										
c. heavier than water										
<b>B. Percent Solid Phase</b>										
Balance ID										
1. Weight of filter (g)										
2. Weight of subsample										
a. gross weight (g)										
b. tare weight (g)										
c. net weight (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3. Weight of filtrate										
a. gross weight (g)										
b. tare weight (g)										
c. net weight (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
d. density of filtrate (g/mL)										
4. Total weight wet solids (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5. Weight percent solids (wet) (%)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
6. Weight percent solids (dry)										
a. gross dry weight 1 (g)										
b. gross dry weight 2 (g)										
c. percent dry solids (%)										
d. Oven Temp (observed) (°C)										
Thermometer Correction Factor										
Oven Temp Actual (°C)	0	0	0	0	0	0	0	0	0	0
7. Vol. of initial aqueous filtrate (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
8. Vol. of initial organic filtrate (mL)										
<b>Comments:</b>										
$\text{(Weight percent dry solids, B.6c)} = 100 \times \frac{\text{(Gross dry weight 2 or 1, B.6.b or B.6.a if B.6.a is blank)} - \text{(Weight of filter, B.1)}}{\text{(Weight of subsample, B.2.c)}}$										
$\text{Net Weight of Subsample, B.2.c} = \text{(gross weight, B.2.a)} - \text{(tare weight, B.2.b)}$										
$\text{Net Weight of Filtrate, B.3.c} = \text{(gross weight, B.3.a)} - \text{(tare weight, B.3.b)}$										
$\text{Total weight wet solids, B.4} = \text{(Weight subsample, B.2.c)} - \text{(Weight filtrate, B.3.c)}$										
$\text{Weight percent wet solids, B.5} = 100 \times \text{(Total weight wet solids, B.4)} / \text{(Weight of subsample, B.2.c)}$										
$\text{(Vol of initial filtrate, B.7)} = \text{(Weight of filtrate, B.3.c)} / \text{(Density of filtrate, B.3.d)}$										

**Attachment 9  
 TCLP Worksheet No. 2**

Analyst: 0		DV-IP-0012											
		TCLP Worksheet No. 2											
		Selection of Leach Fluid											
Login No.		Sample No.											
Sample No.													
<b>C. Leach Fluid Determination-</b> Does not apply to determination of volatile organic components or SPLP.													
1. Particle size reduction? (<1mm) Yes/No													
If yes, write NCM describing how.													
2. Balance ID													
3. Sample weight, 5.0 +/- 0.1g													
4. Add 96.5 (+/- 2% or 94.57mL to 98.43mL)													
a. Pipette ID or Grad Cylinder ID													
5. Initial pH (after 5 min. mixing time)													
a. Start Time for Mixing													
b. Stop Time for Mixing													
c. pH reading after mixing													
6. If pH > 5.0, then add 3.5 mL 1N HCL & mark "X"													
a. HCL Lot# used													
b. Pipette ID													
7. Secondary pH (after 10min at 50C to 55C)													
a. Thermometer ID													
Thermometer Correction Factor													
b. Start Time													
c. Start Temperature (Observed)													
Start Temperature (Actual)													
d. Finish Time													
e. Finish Temperature (Observed)													
Finish Temperature (Actual)													
f. pH reading after heating (temperature corrected)													
<b>D. Selection of Leach Fluid</b>													
1. If pH from C.5. or C.7.f. is <5.0 use Leach Fluid #1													
2. If pH from C.7.f. is > 5.0, use Leach Fluid #2													
3. SPLP Fluid 1: Soils- East of the Mississippi River; Wastewaters; or Wastewaters													
4. SPLP Fluid 2: Soils- West of Mississippi River													
5. SPLP Fluid 3: If VOCs or Cyanide containing wastes.													
6. X if filtrate and fluid are miscible													

**Attachment 10  
 TCLP Worksheet No. 3**

Analyst: 0										
DV-IP-0012 TCLP/SPLP Worksheet No. 3 Bottle Leach Procedure for Metals and Semi-Volatile Organic Components										
										
Login No.										
Sample No.										
<b>E. Determination of Sample Size</b>										
1. Particle size reduction? Yes/no										
If yes, write NCM describing how.										
2. Balance ID										
3. Weight of wet solids after filtration (g)										
<b>F. Determination of Amount of Leach Fluid</b>										
<b>Fluid Type from Wksht 2</b>										
	#VALUE!									
1. TCLP Fluid 1 Lot #										
2. TCLP Fluid 2 Lot #										
3. SPLP 1 (East) Lot #										
4. SPLP 2 (West) Lot #										
5. pH of leach fluid										
6. Vol of Fluid = wet solids x 20 (mL)										
<b>G. Record of Leach - leach period is 16 to 20 hours</b>										
1. Rotator checked to be rotating between 28 and 32 RPM?										
2. Leach start date and time										
3. Leach stop date and time										
4. Room temperature										
Thermometer Correction Factor										
a. Temp Min (Observed) (°C)										
b. Temp Max (Observed) (°C)										
c. Temp Min (Actual) (°C)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
d. Temp Max (Actual) (°C)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
5. pH of leachate										
Was the sample multiphasic?	#DIV/0!									
6. Volume of leachate (mL)										
a. Oil recovered from leachate (mL)										
7. Volume of initial filtrate for recombination (mL)	#DIV/0!									
8. Combined initial filtrate + leachate (mL)	#DIV/0!									
<b>COMMENTS:</b>										
Volume of Fluid, F.4) = (Weight of wet solids, E.3) X 20 (Vol. of Initial Filtrate for Recombination, G.7) = $\frac{(\text{Solids Leached, E.3}) \times (\text{Leachate Recovered, G.6})}{(\text{Tot. Wet Solids, B.4}) + (\text{Fluid Added, F.6})} \times (\text{Initial Filtrate, B.7})$										

Attachment 11  
 TCLP Worksheet No. 4

Analyst: 0		DV-IP-0012								
		TCLP/SPLP Worksheet No. 4								
Login No.:		ZHE Leach								
Sample No.:										
<b>H. Determination of Amount of Leach Fluid</b>										
1. Balance ID										
2. ZHE vessel number										
3. Weight of material added to ZHE (g) "X" if there was headspace in container.										
4. Weight of filtrate in syringe										
a. gross weight (g)										
b. tare weight (g)										
c. net weight (g)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
5. Volume of filtrate in syringe (mL)										
6. Wet solids in ZHE (g)										
7. Weight of fluid to add (g)	0	0	0	0	0	0	0	0	0	0
a. TCLP Fluid 1 Lot #										
b. SPLP Fluid 3 Lot #										
c. pH of Blank Fluid										
8. Percent Wet Solids (%)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
<b>I. Record of ZHE Leach - the Leach period is 16 to 20 hours.</b>										
1. Leak Check										
a. Reading #1 (psi)										
b. Reading #2 (psi)										
2. Rotator checked to be rotating between 28 and 32 RPM?										
3. Leach start date & time										
4. Leach stop date & time										
5. Room temperature										
Thermometer Correction Factor										
a. Temp Min (Observed) (°C)										
b. Temp Max (Observed) (°C)										
c. Temp Min (Actual) (°C)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
d. Temp Max (Actual) (°C)	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
6. <input checked="" type="checkbox"/> if still under positive pressure after leaching										
7. Volume of leachate recovered (mL)										
a. Volume of oil recovered after leaching										
8. Vol. of initial aqueous filtrate for recombination										
a. Calculated from Worksheet 1 (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
b. Calculated from Worksheet 4 (mL)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
<small>                 Net Weight of Filtrate, H.4.c) = (Gross weight, H.4.a.) - (Tare weight, H.4.b.)                  (Percent Wet Solids, H.8) = 100 X [(Wet Solids in ZHE, H.6) / (Weight of material added to ZHE, H.3)]                  Wet Solids in ZHE, H.6) = (Weight of material added to ZHE, H.3.) - (net weight of filtrate, H.4.c.)                  (Vol of Filtrate for recombination, I.7.b) = (Vol of Leachate Recvd, I.6) X (Weight of Filtrate, H.4.c) / (Vol Fluid Added, H.7.)                  Weight of Fluid to add, H.7.) = (Wet Solids in ZHE, H.6) X 20                  (Vol of filtrate for recombination, I.7.a) = [(Wet Solids in ZHE, H.6.) / (Tot Wet Solids, B.4)] X [(Vol Leachate Recvd, I.6.) / (Vol Fluid Added, H.7.)] X (Vol Filtrate, B.7.)             </small>										
COMMENTS:										

## Attachment 12

### How to Batch TCLP and SPLP:

<b>1311_T</b> (Organics) <b>1311T_Hg</b> (Mercury) <b>1311T_M</b> (Metals)	<b>1312_E</b> (Organics) <b>1312_E_Hg</b> (Mercury) <b>1312_E_M</b> (Metals)	<b>1312_W</b> (Organic) <b>1312_W_Hg</b> (Mercury) <b>1312_W_M</b> (Metals)	<b>1311_Z</b> (ZHE)
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#### Overview

The pre-prep methods listed above are specific to the analytes requested, but it is not necessary to batch them all separately. Above, the methods are placed in boxes to indicate which methods can be batched together, with one exception: *SPLP 8260s will be logged with 1312\_E or 1312\_W, which is the same leach method used for organic bottle preps so not all 1312\_E can be batched together and not all 1312\_W can be batched together.*

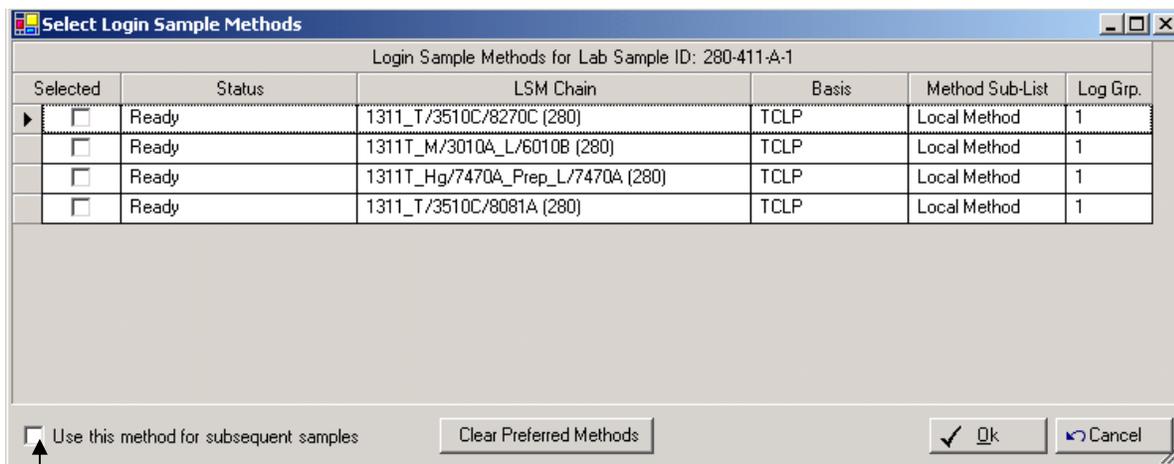
If one sample is logged in for TCLP 8270C, TCLP 8081B, TCLP 8260, TCLP 6010B, and TCLP 7470A, the sample will show up on the Organic Extractions backlog 5 times for TCLP, (once for each analytical method), and twice for 3510C.

Record Status	Status	A-Status	HT Expires	Rush	Method	A-Method	Job Number	Lab Sample ID	Container Matrix
Active	Ready	Active	1/23/2010 11:59	<input type="checkbox"/>	1311_T	8081A	280-J411-1	280-411-1	Solid
Active	Ready	Active	1/23/2010 11:59 PM	<input type="checkbox"/>	1311_T	8270C	280-J411-1	280-411-1	Solid
Active	Ready	Active	1/23/2010 11:59	<input type="checkbox"/>	1311_Z	8260B	280-J411-1	280-411-1	Solid
Active	Ready	Active	2/6/2010 11:59	<input type="checkbox"/>	1311T_Hg	7470A	280-J411-1	280-411-1	Solid
Active	Ready	Active	7/8/2010 11:59	<input type="checkbox"/>	1311T_M	6010B	280-J411-1	280-411-1	Solid
Active	Wait	Active	1/23/2010 11:59	<input type="checkbox"/>	3510C	8081A	280-J411-1	280-411-1	TCLP Leach
Active	Wait	Active	1/23/2010 11:59	<input type="checkbox"/>	3510C	8270C	280-J411-1	280-411-1	TCLP Leach

For the sample above, we would leach the sample in a glass bottle for the organics and metals and we would also do a ZHE leach. Therefore there will be 2 leach batches.

#### Simple Steps

1. Run the OP - TCLP backlog. This backlog is sorted by sample ID so samples logged for multiple extraction and analytical methods will be grouped by sample. Pull the samples from the walk-in cooler and take custody of the samples.
2. Use the TCLP spreadsheets in EXCEL to determine blank fluid for each sample. Once the leach fluid has been determined, you will know what samples can be batched together. You can not put samples with different leach fluids in the same batch.
3. Open Analyst Desktop and select Create Batch from Scratch
4. Your Batch Notes will appear, but we are not going to use the Batch Notes. Instead all of our data will be recorded in the TCLP spreadsheets in EXCEL.
5. Scan your samples into the batch. If your samples are logged in for more than one of the leach methods, a window will appear called "Select Login Sample Methods".



6. Select all LSM Chains that include the leach preps. If all of the samples that you want to batch together are logged in for the same methods, you can click the box in the lower left-hand corner that says “Use this method for subsequent samples”. Then click “OK”.  
**NOTE:** Be careful when clicking the “Use this method for subsequent samples” box. If you click this and the subsequent samples have more methods than the ones listed in the LSM box, they will not be included in the batch. You can check this in Step 10 below and fix it there if there is something wrong.
7. If your batch is for TCLP Fluid #1 or SPLP East Fluid, create a “LB” for the Leach Blank. If your batch is for TCLP Fluid #2 or SPLP\_West Fluid, create a “LB2” for the Leach Blank. If your batch is for water TCLP samples, water SPLP samples, or SPLP ZHE samples, then you are using reagent water as your blank fluid and create a “LB3” for your Leach Blank. There will be no other QC here at this point unless a client has requested MS/MSD on a sample. If that is the case, add the MS/MSD to the leach batch, but it does not get spiked before the leach.
8. Go to the Sample List tab. Here you will see that if the sample was logged in for more than one method chain, the sample will be listed here multiple times – one for each method chain. It is a good idea to check your backlog against the Sample List tab to make sure that all of your method chains that were listed on the backlog are in the batch. If they are not, right click on the sample and click on “Select LSM” to add the missing tests into the batch.
9. Go to the Worksheet tab. We won’t be using the fields here to record our data because the calculations are not locked. We will use the TCLP spreadsheet instead. **But we will have to enter the Leach Fluid type or else our spreadsheets will not get into the raw data. Scroll all the way over to the right and enter “T1”, “T2” “Milli-Q”, “SE”, “SW” or “S3”.**
10. We will use a different status to indicate where the samples are.
  - a. A status of “Batched” or “1<sup>st</sup> Level Review” means the blank fluid determination is done, samples are tumbling.
  - b. A status of “2<sup>nd</sup> Level Review” means that the samples have completed the leachate and have been filtered.
11. Once all steps in the procedure are complete, save the TCLP worksheet in EXCEL. Then print it to pdf and save it in the same directory as the EXCEL file. **When you print it to pdf, be sure to select “Entire Workbook” so that all worksheets will be in the pdf.**
12. Go into the TALS batch and click on the documents button. Right click and from the menu select “Change Document Type”, and then select “External Prep Worksheet”. Attach the pdf of the EXCEL spreadsheet and the pdf of the LogTag Summary to the TALS batch.

## Attachment 13 Instruction Manual for pH Meter

### pH Technique

### pH Calibration

1. Prepare the electrode according to the electrode user guide.
2. In the setup mode, select the buffer set (*USA* or *EU-D*) that will be used for the automatic buffer recognition feature.
3. In the measurement mode, press  until the arrow icon points to the top line, press  until the **pH** icon is shown and press  to begin the calibration.
4. Rinse the electrode, and ATC probe if being used, with distilled water and place into the buffer.
5. Wait for the **pH** icon to stop flashing.
  - a. Automatic buffer recognition – When the **pH** icon stops flashing the meter will display the temperature-corrected pH value for the buffer.
  - b. Manual calibration – When the **pH** icon stops flashing the meter will display the actual pH value read by the electrode. Press  until the first digit to be changed is flashing, press  /  to change the value of the flashing digit and continue to change the digits until the meter displays the temperature-corrected pH value of the buffer. Once the pH buffer value is set, press  until the decimal point is in the correct location.
6. Press  to proceed to the next calibration point and repeat steps 4 and 5 or press  to save and end the calibration.
7. The actual electrode slope, in percent, will be displayed in the main field and *SLP* will be displayed in the lower field.
  - a. For a one point calibration, press  and  /  to edit the slope and press  to return to the measurement mode.
  - b. For a two or more point calibration, the meter will automatically proceed to the measurement mode after the slope is displayed.

**Attachment 14**  
**SPLP worksheet for correcting fluid pH**

**Dilution of Wrong SPLP pH with Water**

	Inputs	Units	Note
pH of solution	1.29		Make sure is below Target pH
pH of Water	5.23		Elga pH is usually 5.25
Target pH	4.2		NOT pH 7
Target Volume (L)	45	L	
		Units	
Required Volume of Water	44.95	L	
Required Volume of Solution	0.05	L	

EXAMPLE



**TestAmerica Denver**

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## Title: ACID DIGESTION OF SOLIDS [Method EPA 3050B]

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## 1.0 **Scope and Application**

- 1.1 This is a strong acid digestion procedure for the preparation of sediments, sludge, soils, and other types of solid materials by EPA Method 3050B for analysis by inductively coupled plasma atomic emission spectroscopy (ICP) or inductively coupled plasma-mass spectrometry (ICP/MS).
- 1.2 Method 3050B is designed to determine the concentration of “environmentally available” metals, and is not a true “total metals” digestion (see discussion below). The procedure is used primarily for hazardous waste characterization and other Resource Conservation and Recovery Act (RCRA) compliance testing.
- 1.3 The elements approved for Method 3050B are shown in Table I. The source method also mentions that other elements may be prepared by the method if the quality control requirements are met. The complete list of elements routinely included in this procedure by TestAmerica Denver is shown in Table II.
- 1.4 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

## 2.0 **Summary of Method**

A representative 1 to 2 gram portion of sample is digested with two cycles of nitric acid additions, followed by hydrogen peroxide digestion. For ICP analysis, the sample is also refluxed with hydrochloric acid. The resulting solution is filtered and diluted to 100 mL with reagent water. For the Incremental Sampling Method, 10 g of sample is used and brought to a final volume of 500 ml.

## 3.0 **Definitions**

- 3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.
- 3.2 **Total Metals** - Although Method 3050B is often referred to as a “total metals” digestion, it is important to understand that there are many compounds formed from these elements that are not efficiently dissolved using this digestion procedure. It is more accurately termed a strong acid digestion procedure. The limitations are discussed further in Section 4 (Interferences) below. The method itself states, “This method is not a total digestion technique for most samples.” There are a variety of total digestion procedures used for metal assay, geochemical analysis, etc., that involve more vigorous digestions than 3050B.
- 3.3 **Preparation Batch** - A group of up to 20 samples that are of the same matrix and are processed together using the same lots of reagents and standards. The minimum QC elements in a batch are outlined in Section 9.
- 3.4 **Reagent Water** – Water that is free of the analytes of interest. In the Metals group, reagent water is obtained from a Barnstead E-Pure water purification system.

- 3.5 Other quality control terminology used in this procedure is based on SW-846, and is defined in the glossary section of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*.

#### 4.0 Interferences

- 4.1 There are common compounds formed by the elements of interest (e.g., barium sulfate, beryllium oxide, silicon dioxide, crystalline silicates, titanium dioxide, etc.) that are not efficiently dissolved using this EPA approved procedure.
- 4.2 Silicon or silica are occasionally requested as part of the Method 3050B digestion. However, this digestion will include only acid-soluble silicon, and will not dissolve crystalline silica. The analysis is for silicon, but the final result is sometimes expressed as silica rather than silicon.
- 4.3 Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP-MS procedure that includes HCl as the final digestion acid. See Section 11.12 of this SOP.
- 4.4 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., powdered gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. See Attachment 1 for more information regarding contaminant control.
- 4.5 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.
- 4.6 For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.
- 4.7 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrix materials may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.
- 4.8 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals or conversion of metals to insoluble forms. For example, antimony is easily lost by volatilization from hydrochloric media. If this occurs the sample must be re-prepared.
- 4.9 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.
- 4.10 Samples Requiring Additional Digestion Reagents

A few examples of types of samples that might require additional digestion reagents follow. It is very important to note situations where samples are not

behaving normally. However, do not assume that adding additional reagents will be acceptable for the project, even if it is obvious that the digestion will be incomplete without it. The situation must be discussed with the project manager and documented in a Nonconformance Memo (NCM), whether or not the variations suggested in the following examples are approved.

- 4.10.1 Samples with high organic content may require additional nitric acid and/or hydrogen peroxide for a thorough digestion, but these oxidizing reagents should be added very carefully to avoid violent reactions.
- 4.10.2 Samples with high concentrations of metal in the elemental form or refractory oxides may require additional hydrochloric acid for a thorough digestion. As an example, blasting sand used to remove paint from the hull of ships typically consists of 30% cupric oxide. Following 3050B exactly will produce results as low as 0.1% without additional hydrochloric acid. Increasing the volume of hydrochloric acid can produce results approaching the true copper concentration. Samples that appear to have nonstandard matrices or have visible metal particles should be documented in an NCM.
- 4.10.3 Highly alkaline materials may require larger volumes of acid than specified in this procedure.
- 4.10.4 If the use of extra digestion reagents is approved, the same volume of reagents must be added to all field samples and QC samples in the batch. Usually the method blank results will not be elevated. To ensure that the QC sample results accurately reflect sample results, the QC samples must be treated exactly like the samples.

## 5.0 **Safety**

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 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, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

### 5.3 **Specific Safety Concerns or Requirements**

5.3.1 Samples that contain high concentrations of carbonates or organic materials or samples that are at elevated pH can react violently when acids are added. If any solid sample appears to be a chemical substance rather than an environmental sample, consult with the group supervisor or the Project Manager (PM) before adding acid.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and

reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

#### 5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

Material <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and Symptoms of Exposure
Hydrogen Peroxide, H <sub>2</sub> O <sub>2</sub>	Oxidizer Corrosive Poison	1 ppm TWA 1.4 mg/m <sup>3</sup> TWA 75 ppm IDLH	Contact with other materials may cause fire. Eye contact may result in permanent eye damage. Causes eye and skin burns. Corrosive: May cause severe respiratory tract irritation. Harmful if swallowed, may cause digestive tract irritation or burns.
Nitric Acid, HNO <sub>3</sub>	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, HCl	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.

## 6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

### 6.1 Instrumentation

**6.1.1** Top-loading balance capable of accurately weighing to the nearest 0.01 grams.

**Note:** Balances are serviced annually and the accuracy checked daily using three standard masses. See SOP DV-QA-0014 for details.

**6.1.2** Digestion "Hot Block" or equivalent heating device capable of maintaining a temperature of 90-95 °C. The Hot Block temperature must be monitored separately for each unit. The temperature of each Hot Block is checked by placing a calibrated thermometer through a cap on a digestion tube that is partially filled with water. The water in the tube must be high enough to cover the thermometer past the minimum immersion line. The temperature is directly recorded in the Batch Information area in the TestAmerica LIMS (TALS).

### 6.2 Supplies

**6.2.1** Thermometers (non-mercury liquid filled or digital) that cover a temperature range including 80-110 °C with clearly visible 1 °C increments.

**Note:** Thermometers are calibrated before use and periodically as described in SOP DV-QA-0001.

**6.2.2** Disposable digestion tubes, with volume accuracy verified to  $\pm 3\%$  gravimetrically prior to use. See SOP DV-QA-0008.

**6.2.3** Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

**6.2.4** Whatman 541 (acid washed) filter paper, or equivalent.

**6.2.5** Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

**6.2.6** Syringes or equivalent filtration apparatus.

**6.2.7** Disposable plastic funnels.

- 6.2.8 Disposable wooden spatulas for subsampling.
- 6.2.9 Centrifuge, capable of at least 2,000 rpm.
- 6.2.10 Graduated cylinders, 100 mL and 500 mL, capable of  $\pm 3\%$  accuracy.
- 6.2.11 Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

**Note:** Mechanical pipettes are calibrated before use as described in SOP DV-QA-0008.

- 6.2.12 Class A volumetric flasks.
- 6.2.13 pH indicator strips (pH range 0 – 6).

### 6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

## 7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

- 7.1 Reagent water – Millipore DI system or equivalent, 10-18.2 megohm-cm. See SOP DV-QA-0026 for daily water monitoring procedure.
- 7.2 Nitric acid ( $\text{HNO}_3$ ), concentrated - Trace metal grade or better.
- 7.3 Nitric acid ( $\text{HNO}_3$ ), 5% - Add 50 mL of concentrated  $\text{HNO}_3$  to approximately 900 mL of reagent water and dilute to 1 liter.
- 7.4 Hydrochloric acid ( $\text{HCl}$ ), concentrated - Trace metal grade or better.
- 7.5 30% Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) - Reagent grade used for ICP analysis.
- 7.6 30% Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) – Ultra pure used for ICP-MS analysis.
- 7.7 Glass beads,  $\leq 1$  mm diameter, washed with aqua regia (for DoD projects).

**7.8 Standards**

**7.8.1** All standards must be NIST traceable. Unless purchased directly from NIST, the accuracy of each standard is verified before the initial use, as described in SOP DV-QA-0015.

**7.8.2 Storage and Shelf Life of Metal Standards**

**7.8.2.1** Standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. They are stored at room temperature.

**7.8.2.2** 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.

**7.8.3 LCS and MS Spike Solutions**

**7.8.3.1** ICP and ICP/MS spike solutions are purchased as custom-made solutions from a commercial vendor at ready-to-use concentrations. No further dilutions are needed.

**7.8.3.2** If a non-routine element is required that is not contained in the custom-made solution, single-element solutions from a commercial vendor may also be used.

**7.8.3.3** Intermediate standards prepared in the laboratory may be used for spiking as long as the procedures for standard recording and verification outlined in SOP DV-QA-0015 are followed.

Typical LCS and MS/MSD spike standard concentrations are shown below. Analysis	Standard	Elements	Conc. (mg/L)
ICP	ICP SPK 3A	Ag, Be, Cd Cr Cu Co, Mn, Ni, Pb, V, Zn As, Fe, Li, Sr, Th Al, Ba, Bi, Se, Tl, U P Ca, K, Mg, Na	5 10 20 25 50 100 200 1,000 5,000
ICP	ICP SPK 2B	Sb, Zr B, Mo, Ti Sn, S Si (SiO <sub>2</sub> )	50 100 200 1,000 (2,140)

Typical LCS and MS/MSD spike standard concentrations are shown below. Analysis	Standard	Elements	Conc. (mg/L)
ICP-MS	MS CALSTD-1	Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Th, Tl, U, V, Zn	20
ICP-MS	MS spike 2	Mo, Sb, Sn, W, Zr Al, Fe	20 200

**Note:** ICP or ICP-MS digestions may select different combinations of spikes in order to satisfy client requests. All spikes used for sample digestion will be recorded in the Reagent module in TALS.

## 8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2 Soil samples do not require chemical preservation, but are stored at  $\leq 6$  °C until the time of analysis.

Matrix	Sample Container	Min. Sample Size	Preservation <sup>1</sup>	Holding Time <sup>2</sup>	Reference
Soils	Glass	3 grams	Cool $\leq 6$ °C	180 Days	N/A

<sup>1</sup> Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot for both analyses must be refrigerated.

<sup>2</sup> Inclusive of digestion and analysis.

## 9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0

unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

**9.1.3** Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

**9.1.4** Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

**9.2** Preparation batches may consist of up to 20 field samples. Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

### **9.3 Minimum QC Requirements**

Each preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. Note that some programs require an unspiked duplicate sample in place of or in addition to the duplicate matrix spike. Be sure to check special instructions in TALS. If clients specify specific samples for the MS and MSD, the batch may contain multiple MS/MSD pairs.

#### **9.3.1 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. Soil method blanks are prepared by taking 5 mL or 5 g of reagent water through the procedure described in Section 11. Add 1.0 g of prewashed glass beads to the blank if required by the client to better simulate a solid matrix.

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.

**Acceptance Criteria:** Criteria for the acceptance of blanks are contained within the individual analytical method SOPs.

**Corrective Action:** If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be re-digested and reanalyzed.

### 9.3.2 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS contains reagent water that is spiked with all the analytes of interest and is carried through the entire analytical procedure. A duplicate LCS (LCSD) must be prepared when there is insufficient sample volume to perform an MS/MSD. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Add 1.0 g of prewashed glass beads to the LCS if required by the client to better simulate a solid matrix.

The spike solutions described in Section 7.8.3 are used to prepare LCSs as follows:

- Routine ICP: Add 1.0 mL of spike
- DoD ICP: Add 1.0 mL of spike to 1.0 g of glass beads
- Routine ICP-MS: Add 1.0 mL of spike
- DoD ICP-MS: Add 1.0 mL of spike to 1.0 g of glass beads

The resulting spike concentrations for each element are given in Table 2 and Table 3.

Incremental Sampling Method LCSs are spiked with 5 ml of spike.

**Acceptance Criteria:** Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs.

**Corrective Action:** When LCS results fail to meet control limits, the LCS and all associated samples in the batch must be re-prepared and reanalyzed.

### 9.3.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a second aliquot of a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Samples identified as field blanks cannot be used for MS/MSD analysis. The MS/MSD results are used to determine the effect of a

matrix on the precision and accuracy of the analytical process.

The spike solution described in Section 7.7.3 is also used to prepare matrix spikes, as follows:

- ICP: Add 1.0 mL of spike
- ICP-MS: Add 1.0 mL of spike

The resulting spike concentrations for each element are given in Tables II through IV. Incremental Sampling Method MS/MSD pairs are spiked with 5 ml of spike.

**NOTE 1:** The spike must be added after the sample aliquot but before any reagents.

**NOTE 2:** This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD pair is not available, an LCS and LCSD are used to measure precision.

## 10.0 Calibration

Not applicable. This SOP addresses sample preparation only for subsequent ICP or ICP/MS analysis. Calibration of the measurement system is covered in the SOPs for the determinative methods.

## 11.0 Procedure

**11.1** One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

**11.2** Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

### 11.3 Sample Custody

- 11.3.1** Samples are transferred from the Sample Control group to the Metals group and the transfer is documented using the Sample Transfer function of the Internal Chain of Custody in TALS (see SOP DV-QA-0003 for details).
- 11.3.2** Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be done in a manner to ensure connection with the proper sample.

### 11.4 Subsampling

- 11.4.1** It is not acceptable to simply collect 1.0 g off of the top of the sample. Samples must be mixed and incrementally subsampled to obtain a representative portion. At a minimum, mix by stirring with a disposable wooden spatula. If there is insufficient room in the sample container to allow for proper mixing, refer to SOP DV-QA-0023, *Subsampling*, for directions.
- 11.4.2** Select at least three incremental subsamples from different locations in the original sample and place them in a tared 50 mL digestion tube. The final sample weight should be between 1.0 and 1.5 g. Record the weight to the nearest 0.01 g.
- 11.4.3** Measure additional aliquots for QC samples required in the batch and spike as required (see Section 9 for details).

**NOTE:** When adding glass beads to the Method Blank and LCS digestion tubes, the nominal weight must be entered into the Initial Amount field in TALS. The true weight of glass beads should be recorded in the Notes field on the Worksheet tab in the preparation batch.

### 11.5 Incremental Sampling Method Digestion

For the Incremental Sampling Method approximately 10 g of sample is weighed out by the Organic Prep group following the procedure described in SOP DV-OP-0013. This pre-weighed sample is then delivered to the Metals group for digestion and analysis. The sample weight is recorded on the ISM Worksheet and attached to the incremental sampling batch in TALS. The pre-weighed aliquots are delivered in 125 mL digestion tubes which are ready for spike standards and reagents to be added. The Method 3050B digestion reagents are increased 5x to maintain the same proportions as are used for a 1-2 gram sample. When required, 10 g of glass beads are added to the Method Blank and LCS prior to digestion.

### 11.6 Initial Digestion Cycle with 1:1 Nitric Acid

- 11.6.1** Add approximately 5 mL of reagent water to each digestion tube.
- 11.6.2** Add 5 mL of concentrated HNO<sub>3</sub>.

**11.6.3** After all of the acid has been added to the preparation batch, gently swirl the samples to mix and then place the sample rack on the Hot Block.

**11.6.4** Place a ribbed cover on each tube.

**11.6.5** Heat samples to 90-95 °C, and reflux for 15 minutes without boiling.

**NOTE: DO NOT ALLOW SAMPLES TO BOIL OR GO DRY** during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.

**11.6.6** Remove the samples from the Hot Block and allow them to cool before proceeding with the next step.

**11.6.7** Record the start time, starting temperature, end time, and ending temperature in TALS.

## **11.7 Second Digestion Cycle Using Concentrated Nitric Acid**

**11.7.1** Add 5 mL of concentrated HNO<sub>3</sub>, and replace the ribbed cover.

**11.7.2** Place samples back on the Hot Block and reflux at 90-95 °C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

**11.7.3** If brown fumes are observed, this means that material in the sample is actively being oxidized. There may not be enough HNO<sub>3</sub> acid to complete the oxidation, and there could be violent reaction of the sample with peroxide in the third digestion step. For that reason, it is necessary to repeat the previous two steps until no more fumes are evolved.

**11.7.4** Heat the samples at 90-95 °C for 2 hours.

**11.7.5** Allow the samples to thoroughly cool before proceeding.

## **11.8 Third Digestion Cycle Using Hydrogen Peroxide**

**11.8.1** Add 2 mL of reagent water to each tube.

**11.8.2** Add 3 mL of 30% H<sub>2</sub>O<sub>2</sub> a few drops at a time. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

**11.8.3** Replace the ribbed cover and heat samples until effervescence subsides.

**11.8.4** Allow the samples to cool.

**11.8.5** Continue adding 30% H<sub>2</sub>O<sub>2</sub> in 1 mL increments with warming until

effervescence is minimal or sample appearance is unchanged. If additional peroxide is added to a sample then it must also be added to the method blank and LCS.

**NOTE:** Do not add more than a total of 10 mL of 30% H<sub>2</sub>O<sub>2</sub>. If 10 mL have been added and the samples are still vigorously effervescing, document the situation with an NCM and continue with the digestion.

**11.8.6** Heat the samples at 90-95 °C for 2 hours.

**11.8.7** Allow the samples to cool.

**11.8.8** If samples will be analyzed by ICP, continue on with the fourth digestion step using HCl in Section 11.8. If the samples will be analyzed by ICP-MS, skip the HCl digestion step and go to step 11.10.

#### **11.9 Fourth Digestion Cycle for ICP Using Concentrated Hydrochloric Acid**

**11.9.1** If the samples are being prepared for ICP analysis, add 10 mL of concentrated HCl to the samples in the digestion tubes and cover with ribbed covers.

**11.9.2** Reflux for an additional 15 minutes without boiling.

**11.9.3** Allow the samples to cool.

#### **11.10 Separating Undigested Solids from the Digestion Solution**

**11.10.1** Filter samples through Whatman 541 or equivalent fiber filters into a graduated 125 mL digestion tube whose accuracy is documented to be better than  $\pm 3\%$ .

**NOTE:** In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

**11.10.2** For samples digested by the Incremental Sampling Method use a 500 mL poly bottle that has been measured after measuring out 500 mL of DI water from a graduated cylinder.

**11.10.3** Wash the original digestion tube and ribbed cover with reagent water to ensure quantitative transfer of all of the digestion solution into the new digestion tube.

**11.10.4** Rinse the funnel and filter paper with reagent water to ensure complete sample transfer into the new digestion tube.

**11.10.5** Re-volume sample to 100 mL with reagent water. This must be done volumetrically, rather than by weight. Record the final volume in TALS. For Multi-Incremental samples the final volume is 500 mL.

### 11.11 Documentation and Record Management

The following information must be recorded for each preparation batch. This information is directly entered into TALS.

- Initial sample weight and final digestion volume
- Preparation analyst and date
- Identification of all reagents and standards
- Identification of all measuring and test equipment used (e.g., balances, thermometers, pipettes)
- Glass beads lot number
- Filter paper lot number
- Digestion tube lot number
- Hot Block ID number
- Fume Hood ID number

### 11.12 Alternate Antimony Preparation for Analysis by ICP-MS

- 11.12.1 Weigh out 1.0-1.5 g soil samples according to the procedure in Section 11.3.
- 11.12.2 Add approximately 5 mL of reagent water to each digestion tube.
- 11.12.3 Spike the LCS, LCSD, MS, and MSD with 1.0 mL of the MS spike 2 standard.
- 11.12.4 Add 2.5 mL concentrated HNO<sub>3</sub> and 2.5 mL concentrated HCl to each sample and batch QC.
- 11.12.5 Cover each tube with a watch glass and reflux on hot block at 90-95 °C for 15 minutes.
- 11.12.6 Filter through Whatman 541 or equivalent filter paper into a new 125 mL digestion tube while still hot.
- 11.12.7 Rinse the filter and funnel with 1.25 ml of hot (~95 °C) concentrated HCl.
- 11.12.8 Rinse three times with hot (~95 °C) reagent water (5 mL rinses.)
- 11.12.9 Place the filter paper and soil residue back into the original sample digestion vessel. Add 2.5 mL concentrated HCl, cover and reflux on the hot block for 20 minutes or until paper dissolves.

**11.12.10** Filter through a fresh filter into the original filtrate. Rinse three times with reagent water (5 mL rinses).

**11.12.11** Bring to final volume of 100 mL with reagent water.

## **12.0 Calculations / Data Reduction**

Not applicable. Calculations of final results are described in the determinative analytical SOPs.

## **13.0 Method Performance**

### **13.1 Method Detection Limit (MDL)**

An MDL must be determined for each analyte/matrix prior to the analysis of any samples. See the SOPs for the determinative analysis methods for details.

### **13.2 Demonstration of Capabilities**

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

**13.2.1** Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

**13.2.2** Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

**13.2.3** If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

**13.2.4** Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

**13.2.5** Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

### **13.3 Training Requirements**

The group leader or supervisor is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered in the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See

requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

#### 14.0 **Pollution Control**

- 14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).
- 14.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

#### 15.0 **Waste Management**

- 15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Program*.
- 15.2 The following waste streams are produced when this method is carried out:
- 15.2.1 Aqueous Acidic (Metals) - Corrosive – Waste Stream J
  - 15.2.2 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

#### 16.0 **References**

- 16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996; Method 3050B.
- 16.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.
- 16.3 Department of Defense Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

#### 17.0 **Method Modifications:**

Item	Method	Modification
1	3050B	Method 3050B Section 7.1 states that a 1-2 g aliquot is to be used. The amount specified by TestAmerica Denver in this procedure is limited to 1-1.5 g in order to prevent increased instrument maintenance and sample reruns due to dilutions.

## 18.0 **Figures, Tables, and Attachments**

Table 1: Method 3050B Approved Analyte List for ICP/ICP-MS

Table 2: Soil LCS and MS/MSD Spikes for ICP

Table 3: Soil LCS and MS/MSD Spikes for ICP-MS

Attachment 1: Contamination Control Guidelines

## 19.0 **Revision History**

- Revision dated 31 October 2016
  - Annual Review
  - Update the temperature heating range to 90-95°C where stated in the SOP
  - Removed the reference to AFCEE throughout SOP
  - Added current section 3.1 – reference to QAM for general definitions
  - Restructured and renumbered section 6.0
  - Added initial paragraph to section 6.0 regarding the documentation of equipment IDs
  - Revised the current sections 6.1 and 6.2 to reflect consistent verbiage and formatting as other SOPs
  - Added current section 7.6 Ultra Pure Peroxide reference
  - Added current footnote 1 to the section 8 table regarding soil preservation
  - Re-numbered previous footnote 1 to be footnote 2 to the section 8 table
  - Updated section 9.1 and subsections to reflect current practices and verbiage
  - Re-numbered Notes in section 9.3.3 to be Note 1 and Note 2
  - Added LCSD required when an MS/MSD is not available to sections 9.3.2 and 9.3.3 Note 2
  - Renumbered and updated section 11.1 and 11.2 to reflect current practices and verbiage
  - Added current section 11.2
  - Updated section 13.2 to reflect current practices and verbiage
  - Added Strontium to Table 3
  - Removed Titanium and Zirconium from Table 3
- Revision 8 dated 31 October 2015
  - Annual Review
  - Edited Sections 9.5.1 and 9.5.2 to clarify glass bead requirement
  - Added definition of reagent water
  - Updated Section 11.6.4 and 11.7.6 to reflect current practice
  - Removed Method exception 1 regarding method blank limits as it no longer applies
  - Added detail to training requirements for new analysts Section 13.3
  - Added note to Section 9.5.3 regarding precision requirements
  - Added note to Section 11.3 regarding recording of glass bead weights
- Revision 7 dated 31 March 2015
  - Annual Review
  - In Section 11.7.8 the section referenced was updated to 11.8

- Updated spike standard name to MS spike 2 in Section 11.11.3
  - Formatting and grammar corrections throughout
  - Section 6.4 removed reference to calibrating digestion tubes
  - Section 6.6 changed name of filter paper to match current practice
  - Section 6.14 added to define computer systems used
  - Sections 7.7.3.1 and 7.7.3.2 combined
  - New Sections 7.7.3.2 - 7.7.3.4 added to define spikes used
  - Table of spike names and concentrations added to Section 7.7.3.4
  - Changed LIMS to TALS throughout
  - Section 8.2 changed storage temperature to  $\leq 6$  °C
  - Deleted Section 9.3, duplicated in 13.2
  - Added new Section 9.3 to address federal requirements
  - Rewrote Section 9.5
  - Changed Sections 9.6 – 9.8 to be subsections of the new 9.5
  - Rewrote Section 11.2.1
  - Removed method modification 2 because it referred to the analytical SOP
  - Created new method modification 2 explaining the 1-1.5 g sample aliquot
  - Section 11.3.2 changed required sample aliquot to 1-1.5 g to help avoid targeting
  - Rewrote Section 11.4 to define and explain the Incremental Sampling Method
  - Added new Section 11.5.3 to explain sample mixing
  - Section 11.7.5 added language to note regarding samples that require more than 10 mL of H<sub>2</sub>O<sub>2</sub>
  - Added detail into Sections 11.9.1 – 11.9.5
  - Folded Section 11.10.1 into 11.10
  - Rewrote list of data to be entered into TALS in Section 11.10
  - Rewrote Section 13.2 to match boilerplate
  - Deleted flowcharts Figures 1 and 2
  - Corrected element list in Table 2
- Revision 6 dated 31 March 2014
    - Annual Review
    - Formatting changes throughout document
    - Added to Section 11.7.5 to add additional peroxide to QC if added to samples
    - Updated section number in text to 11.8 in section 11.7.8
    - Added references for DoD QSM
    - Removed Attachment 2
  - Revision 5 dated 04 March 2013
    - Section 7.7.3.1 Added DoD to the glass beads requirement
    - Section 11.11.2 Added that 5ml of water is added to the samples
    - Section 11.11.3 Changed spike name to 200.8 Cal-2
    - Updated spike level to 1.0ml in Table 3
    - Updated work instructions to current revision.
    - Formatting changes throughout document
  - Revision 4 dated 3 February 2012
    - Changed references of Multi-Incremental Sampling to Incremental Sampling Method throughout document
    - Section 2.0 Added reference to Incremental Sampling Method
    - Section 6.4 Added 50 mL digestion tubes

- Added introductory statement to section 7.0 regarding reagent purity
  - Section 7.1 Updated acceptable criteria for the reagent water
  - Section 9.7.2 Added LCS Incremental Sampling Method spike amounts
  - Section 9.8.2 Added MS/MSD Incremental Sampling Method spike amounts
  - Section 11.4 Updated sample amount for Incremental Sampling Method to 1 10g aliquot
  - Section 11.9 Added Incremental Sampling Method final volume
- Revision 3.5, dated 24 August 2011
    - A note has been added to section 9.8.3 for the addition of the LCS/MS spike before reagents.

*Earlier revision histories have been archived and are available upon request.*

**Table 1.**

**Method 3050B Approved Analyte List for ICP/ICP-MS**

<b>Element</b>	<b>Symbol</b>	<b>CAS Number</b>
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

**Table 2.**

**Soil LCS and MS/MSD Spikes for ICP**

<b>ELEMENT</b>	<b>Stock Standard (mg/L)</b>	<b>Sample Spike (mg/kg)</b>	<b>Final Digested Solution (mg/L)</b>
Aluminum	200	200	2.0
Antimony	50	50	0.5
Arsenic	100	100	1.0
Barium	200	200	2.0
Beryllium	5	5	0.050
Bismuth	200	200	2
Boron	100	100	1.0
Cadmium	10	10	0.1
Calcium	5,000	5,000	50.
Chromium	20	20	0.20
Cobalt	50	50	0.50
Copper	25	25	0.25
Iron	100	100	1.0
Lead	50	50	0.50
Lithium	100	100	1.0
Magnesium	5,000	5,000	50.
Manganese	50	50	0.50
Molybdenum	100	100	1.0
Nickel	50	50	0.50
Phosphorous	1,000	1,000	10.
Potassium	5,000	5,000	50.
Selenium	200	200	2.0
Silicon	1,000	1,000	10.
Silver	5	5	0.050
Sodium	5,000	5,000	50.
Strontium	100	100	1.0
Sulfur	200	200	2.0
Thallium	200	200	2.0
Thorium	100	100	1.0
Tin	200	200	2.0
Titanium	100	100	1.0
Uranium	200	200	2.0
Vanadium	50	50	0.50
Zinc	50	50	0.50
Zirconium	50	50	0.5

**NOTE:** Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

**Table 3.**

**Soil LCS and MS/MSD Spikes for ICP-MS**

<b>ELEMENT</b>	<b>Stock Standard (mg/L)</b>	<b>Sample Spike (mg/kg)</b>	<b>Final Digested Solution (µg/L)</b>
Aluminum	200	200	2,000
Antimony	20	20	200
Arsenic	20	20	200
Barium	20	20	200
Beryllium	20	20	200
Cadmium	20	20	200
Chromium	20	20	200
Cobalt	20	20	200
Copper	20	20	200
Iron	200	200	2,000
Lead	20	20	200
Manganese	20	20	200
Molybdenum	20	20	200
Nickel	20	20	200
Selenium	20	20	200
Silver	20	20	200
Strontium	20	20	200
Thallium	20	20	200
Thorium	20	20	200
Tin	20	20	200
Tungsten	20	20	200
Uranium	20	20	200
Vanadium	20	20	200
Zinc	20	20	200

**NOTE:** Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

## **Attachment 1**

### **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 5% HNO<sub>3</sub> according to the procedure described in SOP DV-IP-0005.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

**The following are helpful hints in the identification of the source of contaminants:**

- Yellow pipette tips and volumetric caps can sometimes contain cadmium.
- Some sample cups have been found to contain lead or cobalt.
- New glassware 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.



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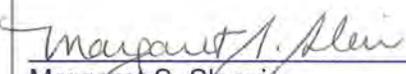
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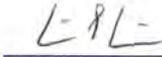
**Title: Polynuclear Aromatic Hydrocarbons by GC/MS Selected Ion Monitoring (SIM)  
[SW 846 Method 8270C and 8270D]**

**Approvals (Signature/Date):**

 2-27-17  
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 28 Feb 17  
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## 1.0 **Scope and Application**

- 1.1 This procedure is a Gas Chromatography/Mass Spectrometry (GC/MS) technique for the analysis of polynuclear aromatic hydrocarbons (PAH) and heterocyclic compounds at the part per trillion (ng/L or ng/kg) level in waters or solids. This procedure follows the general guidelines of EPA Methods 8270C and 8270D for Selected Ion Monitoring (SIM) analysis.
- 1.2 The SIM technique optimizes quantitative information at the expense of qualitative information gained from other methods of analysis. It is important to note that this procedure is intended for the analysis of samples previously characterized by another method such as open-scan 8270C/D. The initial characterization is necessary to avoid misidentification of the parent compounds producing the ions used for this analysis.
- 1.3 In addition, this procedure is appropriate only for sample analytes of interest at less than 10,000 ng/L or 330,000 ng/kg. Samples containing semivolatile organics at concentrations greater than 10,000 ng/L and 330,000 ng/kg should be analyzed by a method designed to detect at higher (part per billion) levels. Samples at these levels may still be analyzed by this procedure, however, extra measurement uncertainty would be introduced because of the sample dilutions that would be required.
- 1.4 This procedure is applicable to water and soil samples. For water samples, 1 liter of water is extracted. It is also possible to extract 250 mL of water and analyze by an LVI (large volume injection) method designed to maintain reporting limits while reducing the initial volume of sample required for extraction. For soil samples, a sample aliquot of 30 g is extracted.

### 1.5 **Analytes, Matrix(s), and Reporting Limits**

The standard list of compounds that can be analyzed by this procedure is shown in Table IV. Typical reporting limits are 100 ng/L for aqueous samples and 5.0 µg/kg for soil samples for the PAH compounds.

## 2.0 **Summary of Method**

### 2.1 **Sample Preparation**

#### 2.1.1 **Aqueous Samples**

Analytes of interest are extracted from water samples using separatory funnel extraction (EPA 3510C or 3510C\_LVI) described in SOP DV-OP-0006. The PAH compounds are extracted from the sample without any adjustment to pH. The concentration of organic extracts is covered in SOP DV-OP-0007.

#### 2.1.2 **Solid Samples**

Solid samples are extracted by sonication (EPA 3550C), which is

covered in SOP DV-OP-0016 or by microwave extraction (EPA 3546) described in SOP DV-OP-0015. The extraction solvent is a 1:1 mixture of methylene chloride and acetone. The concentration of organic extracts is covered in SOP DV-OP-0007.

## **2.2 Instrumental Analysis**

**2.2.1** Quantitation of the extracted compounds is performed by gas chromatography - mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). Routine instrument conditions and the ions used for analysis are shown in Tables I and IV, respectively.

**2.2.2** Development of a successful SIM method requires identifying the ions to be monitored, the ion dwell times, the ions in each group, and the timing for switching between groups. A quantitation ion is selected with a confirmation ion being monitored for identification purposes (see Table IV). Switching times are set where there is adequate resolution (a gap of 1-2 minutes) between peaks. If there is inadequate time between eluting peaks, small retention time shifts may cause peaks to partially or completely disappear as there are changes in the ions monitored. Dwell times will be set by default once the ions per group and the switching times are identified in the data acquisition method. These can be adjusted manually in order to optimize sensitivity as needed.

## **3.0 Definitions**

**3.1** Refer to TestAmerica Denver's Quality Assurance Manual (QAM) and SOP DV-QA-003P for definitions of the quality control terms used in this document.

**3.2** Selected Ion Monitoring - A mass spectrometry technique that provides lower detection level capability by monitoring fewer mass scans for longer periods of time than is done in open-scan methods.

**3.3** Primary Ion Area - The signal chosen for quantitation purposes.

**3.4** Secondary Ion Area - The signal chosen for identification and confirmation purposes.

**3.5** LVI – Large Volume Injection – An analysis method designed to maintain reporting limits while reducing the initial volume of sample required for extraction by increasing the volume of sample extract introduced onto the GC column.

## **4.0 Interferences**

**4.1** Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. The use of high purity reagents and solvents helps to minimize interference problems.

- 4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.
- 4.3 An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound which produces the same ion used for quantitation of one of the PAHs. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not within the specified limits, then interferences may be present. Open scan analysis to identify compounds throughout the mass range is the most reliable assurance against reporting false positives.
- 4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, typically with compound concentrations well in excess of the high calibration standard, the sample analysis that immediately follows the high level sample should be evaluated for carryover. If detections are observed for the compounds that were over the calibration range in the prior sample this sample should be reanalyzed to rule out carryover unless some other objective evidence indicates that carryover is not an issue.

## 5.0 **Safety**

- 5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.
- 5.2 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, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.3 **Specific Safety Concerns or Requirements**
  - 5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately. Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.
  - 5.3.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

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

**5.3.4** There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect the instrument from its source of power.

#### 5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

**Materials with Serious or Significant Hazard Rating**

Material <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm - TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm - TWA 125 ppm - STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
PAH standards can contain all or some of the following: benzo(a)anthracene benzo(b)fluoranthene benzo(k)fluoranthene benzo(a)pyrene chrysene dibenz(a,h)anthracene indeno(1,2,3-cd)pyrene naphthalene	Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen Carcinogen	0.2 mg/m <sup>3</sup> - PEL      10 ppm - PEL	Standards contain low concentrations of compounds known to be or suspected to be carcinogens. All PAH compounds are considered to be hazardous, toxic, and irritants. Some or all are reported human carcinogens, mutagens, and/or teratogens.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

## 6.0 **Equipment and Supplies**

### 6.1 **Instrumentation**

#### 6.1.1 Gas Chromatograph (See Table I for operating conditions)

The analytical system includes a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for split or splitless injection when using capillary columns. Instruments F (Agilent 6890 with a 5973 MSD), G5 (Agilent 6890 with a 5975 MSD), and X4 (Agilent 6890 with a 5973 MSD) may be used for this analysis. Equivalent instruments may be used.

#### 6.1.2 Mass Spectrometer (See Table I for operating conditions)

A mass spectrometer operating at 70 eV (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

6.1.3 A computer system interfaced to the mass spectrometer 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 has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

6.1.4 Please refer to the Master List of Documents, Software, and Hardware (or current revision) located on R:\QA\Read\Master List of Documents for the current software and hardware to be used for data processing.

### 6.2 **Supplies**

6.2.1 All glassware used, both within the scope of this SOP and for the initial sample extraction (see SOPs DV-OP-0006, DV-OP-0008, DV-OP-0007, DV-OP-0015, and DV-OP-0016) must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, and finally with acetone.

6.2.2 Glassware should not be oven dried or heated in a muffle furnace. Successive solvent rinses of the CLLE, separatory funnel, sonication, and Kuderna-Danish glassware are required to minimize low level contamination of samples.

6.2.3 Store glassware inverted or in sealed containers capped with aluminum foil.

6.2.4 Gas-tight syringes, various sizes, and SMI pipettors.

- 6.2.5 Serological pipettes are used for final extract volume measurement.
- 6.2.6 Micro reaction vessels, 1.8 mL vials with Teflon caps, for storing concentrated extracts.
- 6.2.7 Column – A Varian VF-5MS 30-meter fused silica capillary column, 0.5  $\mu\text{m}$  film thickness, 0.25 mm ID, plus 10-meter EZguard, or equivalent.
- 6.2.8 Agilent Ultra Inert splitless single taper liners.
- 6.2.9 Amber crimp cap vials with Sil/PTFE aluminum seals.
- 6.2.10 Hamilton 10  $\mu\text{L}$  autosampler syringes.

## 7.0 Reagents and Standards

### 7.1 Reagents

All solvents are reagent grade or higher unless specified otherwise. See SOPs CA-Q-S-001 and CA-Q-S-001 DV-1 for a description of the program for testing solvents prior to use. The manufacturer expiration applies to all solvents and when not specified by the manufacturer the expiration will be recorded as one year after opening the solvent for use.

- 7.1.1 Methanol, reagent grade.
- 7.1.2 Methylene chloride, reagent grade.
- 7.1.3 Helium gas, 99% + purity.

### 7.2 Standards

Commercial standards are received in flame-sealed ampoules or as neat, 100% concentration solutions. Standards are verified before use. Details concerning verification of standards are given in SOP DV-QA-0015. Stock standards are stored refrigerated at  $\leq 6$   $^{\circ}\text{C}$ . All stock standards must be protected from light. Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier, if the vendor indicates an earlier date. Dilutions or working level standards that are prepared from stock standards are assigned an expiration date according to the earliest expiring stock or one year from the date of preparation, whichever date is earlier.

For the PAH compounds and the additional compounds that are mentioned in this SOP the following stock standards are currently used. **MS-48925** Supelco cat. # 48925 at 1000  $\mu\text{g}/\text{mL}$  (surrogates). **MS-31009** Restek cat. # 31009 SV Calibration Mix #3 at 2000  $\mu\text{g}/\text{mL}$ . **MS-31010** Restek cat. # 31010 SV Calibration Mix #4 at 2000  $\mu\text{g}/\text{mL}$  (has 2-methyl naphthalene). **MS-31853** Restek cat. # 31853 1,4-Dioxane at 2000  $\mu\text{g}/\text{mL}$ . **MS-31995** Restek cat. #31995 8270 CalibrationMix #5 at 2000  $\mu\text{g}/\text{mL}$  (has all PAH compounds including 2 methyl naphthalene). **MS-APP914820X** Accustandard cat. #APP-9-148-20x at 2000  $\mu\text{g}/\text{mL}$  (n-

nitrosodiethylamine). **MS-47643-U** Supelco cat. # CRM47643 8270 Ether/Phthalate mix at 2000 µg/mL. Other vendors and mixes may be substituted for these stocks but an NCM must be written for the SOP deviation.

**7.2.1 GC/MS Tuning Standard**

A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) at a concentration of 50 µg/mL (25 µg/mL for LVI) is prepared by diluting 0.5 mL of the stock to a final volume of 10ml. The current vendor for the tuning standard is Supelco cat. # 47548-U at a concentration of 1000 µg/mL.

**7.2.2 Calibration Standards**

Calibration standards for the initial calibration (ICAL) are prepared at 7 concentrations to cover the calibration range by diluting vendor stock standard solutions using methylene chloride. The standards are prepared directly in autosampler vials by using syringes to deliver the appropriate volumes of stock standard solution, internal standard solution, and methylene chloride. The following tables summarize a typical set of calibration standards:

**MS-SIM SL\_Stk** is a 200 µg/mL calibration substock that is prepared by diluting 0.5 mL of **MS-31009**, **MS-31010**, **MS-31853**, **MS-31995**, (and **MS-APP914820X**) to a final volume of 5 mL.

**Standard Method:** Prepared using a PAH SIM stock standard **MS-SIMSL** with a concentration of 20 µg/mL for levels 4 through 7. The **MS-SIMSL** standard is prepared by diluting 0.2 mL of **MS-48925** (surrogates) and 1 mL of **MS-SIM SL\_Stk** and 0.1 mL of **MS-47643-U** to a final volume of 10 mL. A secondary PAH SIM stock standard **MS-SIMSL Int** prepared by diluting 1 mL of the **MS-SIMSL** to a final volume of 10 mL with a concentration of 2 µg/mL is used to prepare levels 1 through 3:

Vol Stock (µL)	Methylene Chloride (µL)	Internal Standard (µL)	Final Volume (µL)	Conc PAH (µg/mL)
5	495	50	500	0.02
25	475	50	500	0.1
75	425	50	500	0.3
15	485	50	500	0.6
30	470	50	500	1.2
62.5	437.5	50	500	2.5
125	375	50	500	5.0

**LVI Method:** Prepared using a PAH SIM stock standard with a concentration of 20 µg/mL for levels 6 and 7. A secondary PAH SIM stock standard with a concentration of 2 µg/mL is used to prepare levels 1 through 5:

Vol Stock (µL)	Methylene Chloride (µL)	Internal Standard (µL)	Final Volume (µL)	Conc PAH (µg/mL)
1	499	50	500	0.004
5	495	50	500	0.02
15	485	50	500	0.06
30	470	50	500	0.12
60	440	50	500	0.24
12.5	487.5	50	500	0.5
25	475	50	500	1.0

### 7.2.3 Initial Calibration Verification (ICV) Standard (MS-SIM SSV)

A second source initial calibration verification (ICV) standard is prepared using a standard solution that is obtained from a source independent from the source that supplies the standard used for the initial calibration. It is prepared by diluting 30 µL of a substock that is at a concentration of 20 µg/mL to a final volume of 0.5 mL. The final PAH SIM concentration for this ICV standard is 1.2 µg/mL (0.24 µg/mL for LVI).

The substock for **MS-SIMSSV** (above) is prepared by diluting 1 mL of another substock, **MS-HSLB1\_STK**, to 10 mL final volume.

**MS\_HSLB1\_STK** is prepared by diluting 2 mL of **MS-570666.SEC** (Restek cat. # 570666.sec 8270 List 1/Std#1 Mega Mix at 500, 1000, 2000 µg/mL) and 2 mL of **MS-569731SEC** (Restek cat. #769731.sec 8270 List 1/Std #10 at 2000 ug/ml) to a final volume of 10 ml. The final concentration of this stock varies as either 200 µg/mL or 400 µg/mL depending upon the compound.

The final PAH SIM concentration for this ICV standard is 1.2 µg/mL (0.24 µg/mL for LVI).

### 7.2.4 Continuing Calibration Verification (CCV) Standard

A standard with the same analytes and concentrations as the 600 ng/mL (120 ng/mL for LVI) calibration standard. The standard may be from the same preparation as the initial calibration or prepared at a later date.

### 7.2.5 Surrogate Spiking Solutions (8270SIM Surr)

The surrogate spike solution contains neutral surrogates at concentrations of 500 ng/mL in methanol. It is prepared by diluting 0.1 ml

of **8270SurrStkHL** (Restek cat. #567685 at 5000 µg/mL) to a final volume of 1000 ml with acetone. Table II lists the surrogate compounds for the standard list of PAHs.

- 1.0-liter water extractions, add 1.0 mL of the surrogate spike solution
- 250-mL LVI water extractions, add 0.250 mL of the surrogate spike solution
- 30-gram soil sample extractions, add 1.0 mL of the surrogate spike solution

#### 7.2.6 Internal Standard (IS) Solutions (MS-SIM IS)

A 6000 ng/mL solution of the internal standards is prepared in methylene chloride from vendor stock **MS-57604** (Restek cat. #567684 8270 Internal Standard at 2000 µg/mL) by diluting 60ml of this stock to 300 mL final volume. Then 1.5 mL of this stock, **MS-IS**, is diluted to 100 mL to yield the **MS-SIMIS** spiking solution. Table III lists the IS compounds.

To each sample extract, 20 µL of the respective IS solution is added to a 200 µL aliquot of the sample extract for both standard (1 L sample) and LVI extracts.

#### 7.2.7 LCS, MS, and MSD Spike Solution (8270BO-SIMLCS)

A methanol solution containing the requested spike compounds at a concentration of 900 ng/mL each is prepared from vendor stock solution by diluting 0.225 mL of **MS-570666** (Restek cat. #570666 HSL Mega Mix at 1000 µg/mL) to a final volume of 250 mL with P&T methanol. Following are the final sample concentrations of the spiked compounds for the water and solid extractions:

- 1.0-liter water extractions, add 1.0 mL of the spike solution, [PAH] = 900 ng/L
- 250-mL LVI water extractions, add 0.250 mL of the spike solution, [PAH] = 900 ng/L
- 30-gram soil sample extractions, add 1.0 mL of the spike solution, [PAH] = 30 µg/kg

**7.3** All stock and working standards are stored according to the manufacturer's instructions. Dilutions from stocks may not be assigned expiration dates that exceed the stock standard expiration date set by the manufacturer.

## **8.0 Sample Collection, Preservation, Shipment and Storage**

### **8.1 Sample Amounts**

**8.1.1** Water samples are collected in pre-cleaned amber glass bottles fitted with a Teflon-lined cap. To guarantee the ability to meet routine reporting limits, two full bottles of sample should be provided. Additional bottles are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes. For the standard method, each bottle should be 1.0 L; for the LVI method, each bottle should be 250 mL.

**8.1.2** Soil samples are collected in an 8-ounce, pre-cleaned, wide-mouth jar with a Teflon-lined lid.

**8.2** Samples are chilled to a temperature between 0 and 6 °C immediately after collection and shipped via overnight carrier to the laboratory.

**8.3** Samples and excess sample volume must be stored refrigerated at  $\leq 6$  °C from when the log-in process is completed (see SOP DV-QA-0003) to storage after analysis.

**8.4** Water samples must be extracted within 7 days of the time of sample collection, while solid samples must be extracted within 14 days of sampling. Extracts must be analyzed within 40 days from the start of the sample extraction.

## **9.0 Quality Control**

**9.1** The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

**9.1.1** The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

**9.1.2** Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), and Department of Energy (DOE), are described in TestAmerica Denver policy SOP DV-QA-024P, Requirements for Federal Programs. Table 8 details the components of the DoD QSM 5.0 and DoE QSAS 3.0 that are different from TestAmerica Denver's standard procedures, for further details, see SOP DV-QA-024P. Also listed are the variances that TestAmerica is requesting for this analysis; these alternate criteria are only used with project-specific approval.

**9.1.3** Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

**9.1.4** Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

## **9.2 Method Blank (MB)**

A method blank is processed and analyzed with each analytical batch, not to exceed 20 samples. For aqueous samples, the method blank consists of reagent water spiked with surrogates. For soil samples, the method blank is Ottawa sand spiked with surrogates. This sand is mixed with sodium sulfate for extraction by ultrasonication. Method blanks are used to assess whether the laboratory has contributed contamination to the sample analysis process that adversely affects the accuracy of the determination of target analytes. The goal is to have no detectable contaminants in the method blank. However, due to the sensitivity of this analysis, it is not uncommon to detect target analytes at levels above the method detection limit (MDL).

**Acceptance Criteria:** MB results must be less than  $\frac{1}{2}$  the reporting limit.

**Corrective Action:** If the MB exceeds  $\frac{1}{2}$  the RL for any target analyte, then one of the following must apply for acceptance of the batch:

The blank contamination is less than  $\frac{1}{10}$  of the measured concentration of any sample in the associated preparation batch, or

The blank contamination is less than the concentration present in the samples and is less than  $\frac{1}{10}$  of the regulatory limit, or

The same contaminants are not found in the associated samples.

**NOTE:** Positive method blank results below the reporting limit should be evaluated by the analyst for potential impact on sample results at or near the reporting limit.

## **9.3 Laboratory Control Samples (LCS)**

A Laboratory Control Sample (LCS) is processed and analyzed with each analytical batch not to exceed 20 samples. For aqueous samples, the LCS consists of reagent water spiked with the analytes of interest and surrogates. For soil samples, the LCS is Ottawa sand spiked with analytes of interest and surrogates. For ultrasonic extraction, sodium sulfate is added to the reagent sand. The LCS spiking solution is described in Section 7.2.7. LCS results are used to determine whether the analytical system is in control. Depending on project

requirements, a duplicate LCS (LCSD) may be required to assess the precision of the analytical system.

**Acceptance Criteria:** The percent recovery for each requested target analyte in the LCS must fall within the established control limits (found in the LIMS system).

**Corrective Action:** If the percent recovery for any requested analyte in the LCS exceeds the upper control limit and the analyte is not detected in any of the associated samples, then no further action is required, and data are reported with an NCM.

If the percent recovery for any analyte in the LCS exceeds the upper control limit and the analyte is detected in any of the associated samples, then reanalyze the LCS. If similar results are obtained on the second attempt, then investigate and correct any problems. Re-extract and reanalyze the preparation batch.

If the percent recovery for any analyte in the LCS is below the lower control limit, reanalyze the LCS. If similar results are obtained on the second attempt, then investigate and correct any problems. Re-extract and reanalyze the preparation batch.

If re-extraction of samples is not possible or the client requests the samples not be re-extracted, qualify data and explain in a NCM.

#### 9.4 Matrix Spike and Spike Duplicate (MS/MSD)

One matrix spike (MS) sample and one matrix spike duplicate (MSD) sample are prepared and analyzed for each preparation batch. An MS sample is a field sample to which known amounts of the target analytes, as well as the surrogates, have been added. An MSD is a second aliquot of the same sample that is spiked the same as the MS. The MS/MSD spiking solution is described in Section 7.2.7. MS results are used to assess the effects of the sample matrix on the accuracy of the analytical system. The MSD results are used to assess the effects of the sample matrix on the precision of the analytical system. Given the expected variability in sample matrix, the MS/MSD results are applicable to only the sample used to prepare the MS and MSD. MS/MSD results should not be extrapolated to other samples without extensive investigation and characterization to demonstrate similarity between samples. The DoD QSM 5 requires that the MS/MSD be prepared from samples from the same site.

**Acceptance Criteria:** The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at  $\pm 3$  standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD

control limit is set at 3 standard deviations above the mean of the historical data.

**NOTE:** DOD QSM 5 limits apply to projects performed under this program.

**Corrective Actions:** The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as "NC" (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as "NC" (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being

flagged as an over-range measurement (e.g., the E-flag qualifier).

- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

**NOTE:** See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

**NOTE:** Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

## 9.5 Internal Standards

The internal standards listed in Table III and described in Section 7.2.6 are spiked at the same level in all field sample extracts, QC sample extracts, instrument blanks, and calibration standards.

**Acceptance Criteria:** The peak area for each internal standard in each field sample and QC sample extract should be between 50% and 200% of the peak area for the same internal standard in the midlevel standard of the initial calibration.

**Corrective Action:** If the internal standard fails acceptance criteria, then perform the following corrective actions:

- Inspect system for malfunction and correct as needed.
- Reanalyze the affected samples.
- If the interference cannot be corrected for field samples, the earlier analysis is reported with discussion in an NCM.
- If QC samples have internal standard failures that are confirmed by re-analysis, the cause of the failures

must be investigated.

## 9.6 Surrogate Compound Analysis

Surrogate compounds listed in Table II and described in Section 7.2.4 are added to all field and QC samples prior to extraction. Surrogate recoveries are used to assess individual sample matrix effects on sample preparation and analysis.

**Acceptance Criteria:** Surrogate recoveries must fall within established control limits. QC sample results are not acceptable unless the surrogate recoveries for those samples are in control.

**Corrective Action:** Corrective action must be considered for any surrogate failure and may depend on project-specific instructions. Lacking instructions to the contrary the following actions shall be taken:

- Evaluate sample chromatogram and other QC.
- If the surrogate(s) fail in the LCS and/or method blank, then re-prepare and reanalyze all associated samples. Samples may be excepted where the surrogate recovers high in the MB and the MB does not have detection above  $\frac{1}{2}$  of the RL. Likewise, if the surrogate is out of control in the LCS but the LCS compounds recover in control then the samples may be reportable but the program requirements must be checked to see if this is acceptable. In any case an NCM must be written to describe the situation.
- For surrogate failures in field samples, re-prepare and reanalyze the samples, unless matrix interference is evident from earlier analysis or from chromatograms in which case the samples are reported with an NCM.

## 9.7 Instrument QC

### 9.7.1 Instrument Optimization

**9.7.1.1** The GC/MS system must be tuned to meet manufacturer's specifications, using a suitable calibration such as perfluorotri-n-butylamine (FC-43). This is performed through the auto-tune feature in the software. The mass calibration and resolution of the GCMS system is then verified by the analysis of DFTPP prior to the analysis of any standards or samples. In some instances the laboratory will opt to omit the DFTPP. The DFTPP tune check is less useful for SIM analysis than it is for full scan analysis because the DFTPP analysis must necessarily be done in full scan mode. When this check is omitted, the FC-43 check will be performed daily.

**9.7.1.2** The instrument is tuned for DFTPP (decafluorotriphenylphosphine), calibrated initially with a seven-point calibration curve, and verified each 12-hour shift that samples are to be run with one or more continuing calibration verification (CCV) standard(s).

## **9.7.2 Instrument Tuning**

At the beginning of every 12-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table VI) are achieved for DFTPP.

**9.7.2.1** Inject 1  $\mu\text{L}$  of the 50  $\mu\text{g}/\text{mL}$  GC/MS tuning standard (see Section 7.2.1) into the GC/MS system.

**9.7.2.2** The mass spectrum of the DFTPP must be obtained in the following manner: three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is also required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of the DFTPP. Do not subtract part of the DFTPP peak. A procedure compliant with these requirements is programmed into a Macro used to evaluate the DFTPP spectrum. Confirm that all the key  $m/z$  criteria in Table VI are achieved.

**9.7.2.3** If all the criteria are not achieved, the analyst must adjust or retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

## **9.7.3 Initial Calibration (ICAL)**

**9.7.3.1** A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns and source maintenance.

**9.7.3.2** A minimum five-point initial calibration curve must be established for linear fit calibrations (weighted or unweighted). Six points or more are required for second order curve fits. See Section 9.7.4 for Calibration Acceptance Criteria.

- The concentrations of standards commonly used to construct the PAH calibration curve are 20, 100, 300, 600 (often analyzed before the rest of the standards and called the ICIS), 1200, 2500, and 5000  $\text{ng}/\text{mL}$ .

- For the LVI method, the concentrations of standards commonly used to construct the PAH calibration curve are 4, 20, 60, 120 (often analyzed before the rest of the standards and called the ICIS), 240, 500, and 1000 ng/mL.

**9.7.3.3** If the concentration of any target compound in a sample exceeds the calibration range, the extract must be diluted with methylene chloride so that the concentrations of all target compounds fall within the range of the calibration curve, and be reanalyzed. Any samples analyzed immediately following the sample that exceeded the linear range may require reanalysis due to possible carryover from the high-level sample.

**9.7.3.4** Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is supported or adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason can be clearly documented, for example a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 12 hours.

**9.7.3.5** Calculate the response factor (RF) for each analyte for each calibration standard level as described in Section 11.3. Calculate the mean RF and relative standard deviation (RSD) for each analyte.

#### **9.7.4 Calibration Acceptance Criteria and Corrective Action:**

##### **Acceptance Criteria 8270C:**

The RSD of the initial calibration for each analyte of interest must be  $\leq$  35%.

##### **Acceptance Criteria 8270D:**

Refer to Table VII for the acceptance criteria for minimum response factor and RSD. Two target compounds and surrogates may fail to meet the minimum RRF criteria listed in Table VII but must still meet the minimum RRF criteria of 0.010 (excluding compounds with a minimum RRF requirement of 0.010). In addition, two target compounds and surrogates may fail to meet the RSD criteria listed in Table VII but must still meet the maximum RSD requirement of 40%. (excluding compounds

with a maximum RSD requirement of 40%). Refer to SOP DV-QA-024P for requirements for federal programs.

#### **Acceptance Criteria for DoD5:**

The RSD of the initial calibration for each analyte of interest must be < 15%. See SOP DV-QA-024P for further details for QSM 4.2 requirements.

#### **Corrective Actions:**

If these criteria cannot be met, least-squares weighted or unweighted linear regression may be used to establish a calibration function as described in Section 11.4. In this case, the correlation coefficient ( $r$ ) must be greater than 0.995 (equivalent to  $r^2 \geq 0.99$ ) or a second-order regression fit with coefficient of determination (COD,  $r^2$ ) greater than 0.99 may be used. If these linearity criteria are not achieved, verify the standard preparation and instrument conditions, and then recalibrate the instrument. If technical acceptance criteria are not met, it may be necessary to clean the ion source, perform injector maintenance, change the column, or take other corrective actions.

- 9.7.5** In the event that a least-squares regression is used, the analyst should evaluate the bias at the lower portion of the curve. This can be accomplished by re-fitting the low point standard back into the curve. The recalculated concentration should be within  $\pm 50\%$  of the standard's true concentration. If these criteria are not met, the analyst may have to evaluate the concentration range of the standards, or the lower limit of quantitation.

### **9.8 Initial Calibration Verification (ICV)**

The Initial Calibration Verification (ICV) is a second-source, mid-level standard that is analyzed immediately following the initial calibration standards.

**Acceptance Criteria:** The absolute value of the difference between the measured PAH analyte concentration and the true value must be  $\leq 30\%$  or be  $\leq 20\%$  for DoD QSM 4.2 or 5.0.

**Corrective Action:** If the ICV recovery fails, then take the following actions:

- Verify standard preparation, and if incorrect, re-prepare the ICV standard solution.
- If preparation of the ICV standard was correct, then re-prepare the initial calibration standards and recalibrate.

### **9.9 Continuing Calibration Verification (CCV)**

Every 12 hours, the mass spectrometer response for each PAH relative to the internal standard is determined by analyzing a 600 ng/mL calibration standard

(120 ng/mL for the LVI method). The RF for each compound in the continuing calibration verification (CCV) analysis is compared to the RF for that compound in the ICAL.

#### **9.9.1 Acceptance Criteria 8270C**

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must be  $\leq 35\%$ .

#### **9.9.2 Acceptance Criteria 8270D**

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must meet the criteria in Table VII. The compounds must also meet the minimum response factor criteria listed in Table VII. Two target compounds and surrogates may fail to meet the minimum RRF criteria in Table VII (excluding compounds with a minimum RRF requirement of 0.010) but must still meet the minimum RRF criteria of 0.010. In addition, two target compounds and surrogates may fail to meet the difference criteria in Table VII (excluding compounds with a maximum percent difference requirement of  $\pm 40\%$ ) but must still meet the maximum difference requirement of  $\pm 40\%$ . (Refer to SOP DV-QA-024P for requirements for federal programs).

#### **9.9.3 Acceptance Criteria for DoD QSM 4.2 or 5.0**

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must be  $\leq 20\%$  for DoD QSM 4.2 or 5.0.

#### **9.9.4 Acceptance Criteria 8270C & 8270D**

**9.9.4.1** The internal standard response of the CCV must be within 50-200% of the internal standard response in the mid-level (ICIS) of the most recent ICAL sequence.

**9.9.4.2** The internal standard retention time must be within  $\pm 30$  seconds of the internal standard retention time in the corresponding level of the most recent ICAL sequence.

#### **9.9.5 Corrective Action:**

**9.9.5.1** If, for any analyte, the CCV RF does not meet the stipulated acceptance criteria, a five-point calibration curve must be repeated for that analyte prior to the analysis of samples.

**9.9.5.2** If any internal standard retention time in the CCV changes by more than 30 seconds from that of the corresponding level of the most recent ICAL sequence, the chromatographic system must be inspected for malfunctions and corrections made, as required.

## **9.10 Closing CCV (DoD QSM 5.0 only)**

DoD QSM 5.0 requires a closing CCV, injected within 12 hours of the DFTPP injection.

### **9.10.1 Acceptance Criteria**

All reported analytes and surrogates must be within  $\pm 50\%$ .

### **9.10.2 Corrective Action**

Recalibrate and reanalyze all affected samples since the last acceptable CCV

Or

Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails perform column maintenance and recalibrate; then reanalyze all affected samples since the last acceptable CCV.

## **10.0 Procedure**

**10.1** One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

**10.2** Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

### **10.3 Sample Preparation**

#### **10.3.1 Aqueous Sample Extraction and Concentration**

**10.3.1.1** Instructions for the extraction of aqueous samples may be found in SOP DV-OP-0006.

**10.3.1.2** Instructions for the concentration of extracts may be found in SOP DV-OP-0007.

#### **10.3.2 Soil Sample Extraction and Concentration**

**10.3.2.1** Instructions for the ultrasonic extraction of soil samples may be found in SOP DV-OP-0016.

**10.3.2.2** Instructions for the microwave extraction of soil samples may be found in SOP DV-OP-0015.

**10.3.2.3** Instructions for the concentration of extracts may be found in SOP DV-OP-0007.

#### **10.4 Sample Analysis**

**10.4.1** All aliquotting, extract dilutions, and spike additions must be performed in the trace fume hood using equipment dedicated to PAH-SIM analysis. An aliquot of 200ul of each sample extract is placed into a two-milliliter GC/MS autosampler vial. Sufficient volume of extract remains should reanalysis be necessary.

**10.4.2** Prior to analysis, 20 uL of internal standard is added to the sample vial giving a final internal standard concentration of 600 ng/mL (150 ng/mL for LVI) in the extract.

**10.4.3** Representative aliquots are injected into the gas chromatograph/mass spectrometer using similar conditions to those summarized in Table I. The injection volume is 1 µL (5 µL for LVI).

**10.4.4** Whenever an unusually concentrated sample is encountered, it may be necessary to reanalyze the subsequent sample extracts after analyzing an instrument blank to demonstrate that there is no cross contamination.

**10.4.5** The following is a typical analytical sequence:

- Solvent rinses, as needed
- MS tune
- ICAL plus ICV or CCV
- Instrument blank
- MB, LCS
- LCSD (if requested by client)
- Sample extracts
- MS and MSD are interspersed with sample extracts, and usually run after the sample from which they are produced.
- The last sample extract must be injected within 12 hours of the tune.

**10.4.6** The sequence may be altered to accommodate reanalysis or additional instrument blank and calibration evaluations. At a minimum, an instrument blank or a method blank shall be included in the sequence. Refer to QC policy DV-QA-003P for additional details.

**10.4.7** The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses for each analyte as shown in Table IV.

**10.4.8** All compounds detected at concentrations above the method MDL are checked to ensure that the confirmation ion is present at the appropriate ratio.

**10.4.9** All compounds detected at concentrations above the highest calibration standard require dilution and reanalysis. In addition, any samples that were analyzed immediately following a high-level sample should be reanalyzed to rule out carryover from the high-level sample, unless they are preceded by an acceptable instrument blank or the high compound(s) were not detected in the subsequent samples.

#### **10.4.10 Manual Integrations**

**10.4.10.1** Upon completion of the analytical sequence, transfer the raw instrument data to Chrom for further processing. Review the chromatograms to ensure correct assigning of peaks and correct integration of each peak.

**10.4.10.2** Note that certain compounds (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene) may require frequent manual integrations. Special attention must be exercised by the analyst and secondary reviewer for compounds that are commonly mis-integrated in automated software or are manually integrated. If manual data manipulations are necessary, they must be justified and documented. See DV-QA-011P requirements for manual integration.

### **10.5 Troubleshooting and Maintenance**

#### **10.5.1 Daily Instrument Maintenance**

In addition to the checks listed in Appendix B, the following daily maintenance should be performed.

- Clip column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.
- Perform mass calibration as necessary.

#### **10.5.2 Major Maintenance**

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the “topboard” or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

## 11.0 Calculations / Data Reduction

### 11.1 Qualitative Identification

Obtain electronic ion current profiles (EICP) for the primary mass ion and the confirmatory ion for detected compounds. The following criteria must be met to make a qualitative identification:

- 11.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- 11.1.2 The retention time (RT) of unknown peaks must fall within  $\pm 0.2$  minute of the RT for the compound in the daily calibration standard (mid-point ICAL or daily CCV).
- 11.1.3 The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within  $\pm 20\%$  of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library. A compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the mass spectroscopist. Supportive information includes correct relative retention time (RRT) and the presence of the secondary ion, but the ratio falls outside of  $\pm 20\%$  of the primary ion, which may be caused by an interference of the secondary ion.
- 11.1.4 Structural isomers that have very similar mass spectra and less than a 30-second difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if there is a definitive inflection between the two peaks, according to the analyst's judgment. Otherwise, structural isomers are identified as isomeric pairs.

- 11.2 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points* and the public folder *Arizona Calibration Training*.

### 11.3 Average Response Factor Calibration

The following formula is used to calculate the response factor for each analyte of interest relative to the applicable internal standard for each of the calibration standards:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

$A_s$  = Area of the characteristic ion for the target analyte in the calibration standard

$A_{is}$  = Area of the characteristic ion for the internal standard  
 $C_{is}$  = Concentration of the internal standard, (ng/mL)  
 $C_s$  = Concentration of the target analyte in the calibration standard (ng/mL)

The calibration uses the average response factor for each target analyte, which is calculated as follows:

$$\text{average(mean) RF} = \overline{RF} = \frac{\sum_{i=1}^n RF_i}{n}$$

Where:

$RF_i$  = Response factor for the  $i^{\text{th}}$  calibration level  
 $n$  = Number of calibration levels

The standard deviation for the mean RF for each target analyte is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (RF_i - \overline{RF})^2}{n-1}}$$

The relative standard deviation (RSD) for the average response factor for each target analyte is calculated as follows:

$$RSD = \frac{SD}{\overline{RF}} \times 100\%$$

The concentration of each target analyte in the sample extract is calculated using the average response factor that was calculated in the equation above as follows:

$$C_e = \frac{A_e \times C_{is}}{A_{is} \times \overline{RF}}$$

Where:

$C_e$  = Concentration of target analyte in the sample extract, ng/mL  
 $A_e$  = Area of the characteristic ion for the target analyte in the sample extract.  
 $A_{is}$  = Area of the characteristic ion for the internal standard  
 $C_{is}$  = Concentration of the internal standard, (ng/mL)  
 $\overline{RF}$  = Average response factor for the target analyte as determined by calibration

#### 11.4 Linear Least-Squares Regression Calibration (Unweighted)

A linear least-squares regression is performed using the concentration of the target analyte in the calibration standard as the independent variable (x) and the

instrument response as the dependent variable (y). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where:

y	=	instrument response (e.g., peak area)
x	=	concentration of target analyte in calibration standard
m	=	slope of the line
b	=	intercept of the line

For the internal standard calibration, the regression equation is rewritten as follows:

$$\frac{A_s C_{is}}{A_{is}} = m C_s + b$$

Where:

$A_s$	=	Area of the characteristic ion for the target analyte in the calibration standard
$A_{is}$	=	Area of the characteristic ion for the internal standard
$C_s$	=	Concentration of the target analyte in the calibration standard, (ng/mL)
$C_{is}$	=	Concentration of the internal standard, (ng/mL)
m	=	slope of the line
b	=	intercept of the line

The concentration in an unknown extract is then calculated by rearranging the calibration equation as follows:

$$C_e = \frac{\left[ \frac{A_s C_{is}}{A_{is}} - b \right]}{m}$$

Where  $C_e$  is the concentration of the target analyte in the sample extract, and  $A_e$  is the area of the characteristic ion for the target analyte in the sample extract.

The actual sample concentration (C) for each compound is calculated as follows:

$$C = C_e \times \left( \frac{V_e}{V_o} \right) \times DF$$

Where:

C	=	Concentration of the target analyte in the original sample, ng/L (water sample) or ng/kg (solid sample)
$C_e$	=	Concentration of the target analyte in the sample extract, ng/mL
$V_e$	=	Final extract volume, mL.
$V_o$	=	The original volume or weight of the sample that was extracted, L (aqueous sample) or kg (solid sample).
DF	=	Dilution factor, if appropriate.

### **11.5 Additional Regression Calibration Models**

As needed, weighted linear least-squares or second order regressions may be utilized for this analysis. See Corporate SOP CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points* (Attachment 1) and the public folder *Arizona Calibration Training* for calculations and further explanations.

- 11.6** A second-level technical review of the organic data is performed prior to data reporting. This review is performed by a peer or supervisor using the guidelines and checklists detailed in SOP DV-QA-0020.

## **12.0 Method Performance**

### **12.1 Method Detection Limit Study (MDL)**

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

### **12.2 Demonstration of Capabilities**

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

**12.2.1** Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

**12.2.2** Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

**12.2.3** If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

**12.2.4** Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

**12.2.5** Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

### **12.3 Training Requirements**

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

### **12.4 Retention Time Study**

**12.4.1** Expected absolute retention times (RTs) are initially determined by analyzing all target analytes in the open-scan mode. Example RTs are listed in Table V.

**12.4.2** Relative retention times (RRTs) are then calculated for samples in each analytical run based on the RTs found in the continuing calibration verification standard (CCV).

**12.4.3** RTs are re-established after any significant instrument maintenance, including source cleaning and changing columns, or whenever compounds are not adequately detected in CCVs or LCSs.

### **13.0 Pollution Control**

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

### **14.0 Waste Management**

**14.1** All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

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

**14.2.1** Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

**14.2.2** Methylene chloride solvent rinse waste – Waste Stream B

**14.2.3** Expired extract vial waste – Waste Stream A

**14.2.4** Radioactive and potentially radioactive waste must be segregated from non-radioactive and mixed waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

## **15.0 References / Cross-References**

- 15.1** Test Methods for Evaluating Soil Waste Physical/Chemical Methods (SW-846), Third Edition, September 1986, Final update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final update IIB, January 1995; Final Update III, December 1996, Final Update IV January 2008.
- 15.1.1** Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.
- 15.1.2** Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, December 1996.
- 15.1.3** Method 8000C, Determinative Chromatographic Separations, Revision 2, February 2007.
- 15.1.4** Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, February 2007.
- 15.1.5** Method 3510C, Separatory funnel Liquid-Liquid Extraction, Revision 3, December 1996.
- 15.1.6** Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.
- 15.1.7** Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.
- 15.1.8** Method 3546, Microwave Extraction, Revision 0, February 2006.
- 15.2** CLP Statement of work for Multi-Media, Multi-Concentration Organics Analysis, SOM01.2. June 2007.

## **16.0 Method Modifications**

- 16.1** The CLP SOW referenced in 8270D does not require the analysis of DFTPP prior to the analysis of samples. The method relies on the successful analysis of calibration standards to verify acceptable function of the mass spectrometer. TestAmerica Denver utilizes the DFTPP check to identify any operational issues with the mass spectrometer prior to the analysis of the calibration standards. This allows the analyst to identify possible problems independent of the GC. As a result, the laboratory will start the 12 hour clock with the injection of the DFTPP, not the calibration standard as required in the method.
- 16.2** Method 8270C serves as the basis for this SOP, but the method has been modified extensively for low-level analysis using selected ion monitoring (SIM) and optimizing instrument conditions for the low-level analysis. Consequently the sensitivity of the method has been enhanced and it is not uncommon to detect low-level contamination in the method blank at levels well below the limits of detection for the less sensitive GC/MS method. For example, Method 8270C states that the

RSD of the initial and continuing calibration must be less than or equal to 15% and 20% respectively. Due to the low-level nature of the analysis, this SIM procedure allows both of these criteria to be less than or equal to 35%.

**16.3** Method 8270C stipulates qualitative identification based on relative retention time (RRT), which is calculated by dividing the retention time (RT) of the target analyte by the RT of the internal standard. The RRT of the suspected target analyte in the sample extract must be within  $\pm 0.06$  RRT units of the RRT for that analyte in the calibration standard. This SOP stipulates qualitative identification based on an absolute RT. Namely the RT of the suspected target analyte in the sample extract must be within  $\pm 0.2$  minute of the RT for that analyte in the calibration standard. Additionally, the RT for the internal standard in the sample extract must also be within  $\pm 0.2$  minute of the RT for the internal standard in the calibration standard. The criteria used in this SOP are more restrictive than those imposed by the referenced method. For the earliest eluting compounds, the RT for the internal standard is typically 8 minutes. The earliest eluting target analyte must be at a RRT of at least 0.8, which translates to a RT of 6.4 minutes. Assuming a worst-case scenario where the RT of the internal standard is 0.2 minute higher (i.e., 8.2 minutes) and the RT of the target analyte is 0.2 minute lower (i.e., 6.2 minutes), the calculated RRT is 0.76. The total deviation from the expected RRT is 0.04 RRT units, which is smaller than what is allowed by Method 8270C.

## **17.0** Attachments

Table I:	Routine Instrument Operating Conditions
Table II:	Surrogates for Standard List Analysis
Table III:	Internal Standards for Standard List Analysis
Table IV:	PAH Compounds and Ions Used for Analysis
Table V:	Example Retention Times, IS and Surrogate Associations
Table VI:	DFTPP Key Ions and Ion Abundance Criteria for 8270C and 8270D
Table VII:	8270D Relative Response Factor Criteria for Initial and Continuing Calibration
Table VIII:	Specific DoD QSM 5.0 and DoE QSAS 3.0 Requirements for 8270D
Appendix I:	Extended List PAHs
Appendix II:	Suggested Instrument Maintenance Schedule – Mass Spectrometer & Gas Chromatograph
Appendix III:	Mass Spectrometer Settings for Single Ion Monitoring

## **18.0** Revision History

- Revision 11: 28 February 2017
  - Added Section 4.4 to address contamination by carryover.
  - Added details on instruments currently used in Section 6.1.1.
  - Added supplies as Sections 6.2.8-6.2.10.
  - Revised standards information in Section 7.0 to reflect current TALS names in the reagent module and to reflect current practice regarding makeup of solutions.
  - Added clarification for when NCM is written in lieu of reprep/reanalysis for failed LCS.
  - Revised Section 9.4 on MS/MSD to reflect current policy.
  - Added detail to surrogate corrective actions in Section 9.6.
  - Revised volume of IS added in Section 10.4.2 to reflect current practice.
  - Revised Section 12 to reflect current practice.

- Added list of injection volumes by method chain in Table I.
- Editorial and formatting changes throughout.
- Revision 10: 2 September 2015
  - Formatting and editorial changes throughout
  - Updated Section 9.4 on corrective action for MS/MSD to reflect current practice
  - Added requirement for Initial Calibration to be  $\%RSD = \pm 15\%$ , or Linear regression or 2<sup>nd</sup> order to be  $r^2 \geq 0.99$ ; CCV and ICV to be  $\pm 20\%$  to meet DOD 5 criteria in appropriate sections per DOD requirement that these must be explicitly stated in the SOP.
  - Added new Section 9.10 to address closing CCV required by DoD QSM 5.0.
  - Updated references to incorporate SOP on Calibration Curves in Section 11.2 and 11.5.
  - Updated Sections 12.1-12.3 to reflect current practice.
  - Updated Table IV analytes and ions used for analysis
  - Added Appendix III to identify MS Settings for SIM for each compound
- Revision 9: 31 August 2014
  - Added Table 8, Specific DoD QSM 5.0 and DoE QSAS 3.0 Requirements for 8270 C or D
  - Added reference to DoD QSM 5.0
  - Modified the large volume injection (LVI) internal standard concentration to 600ng/mL
  - Added Appendix II, Suggested Instrument Maintenance Schedules
- Revision 8: 31 August 2013
  - Annual Technical Review
  - Added references to analysis by LVI
  - Updated Appendix I to reflect current practice
- Revision 7: 31 July 2012
  - Annual Technical Review
  - Grammatical and formatting changes throughout
  - Updated the quant ion for surrogate terphenyl-d14 to IS#2 in Table V
  - Updated Table 1 to match current GC conditions
- Revision 6.2: 31 August 2011
  - Inserted Section 7.2.4.
  - Revised QC section (Section 9)
  - Inserted paragraph 10.2.10. regarding manual integration
  - Added Section 11.5
  - Revised Section 16.2 regarding calibration criteria
  - Updated prep methods used and inserted prep methods in reference section
  - Annual Technical Review
  - Grammatical and formatting changes throughout

*Earlier revision histories have been archived and are available upon request.*

**Table I: Routine Instrument Operating Conditions**

<b>GC Conditions<sup>1</sup></b>	
Inlet	Split or Pulsed Split at 275 °C Split ratio - 3.1 : 1 Split Flow – 10.4 mL / min
Capillary Column	Varian Vf-5MS, 30 m length, 0.25 mm diam ID, 0.5 µm thickness
Column Mode	Constant flow, 3.4 mL/min
Temperature Program	Initial temp = 55 °C 30 °C/min ramp to 256 °C 4 °C/min ramp to 296 °C 30 °C/min ramp to 340 °C and hold for at least 1 minute past the elution time of the last compound.
Run Time	About 20 minutes with a new column.
Carrier Gas	Helium Purge flow = 25.0 mL/min, 3.00 min Total flow ≈ 31 mL/min
Injection Volume	Injection volume will be 1.0 µL or 5.0 µL depending on the logged method chain. <ol style="list-style-type: none"> <li>1. 8270C_SIM/3510C = 1.0 µL</li> <li>2. 8270C_SIM/3510C_LVI = 5.0 µL</li> <li>3. 8270D_SIM/3510C = 1.0 µL</li> <li>4. 8270D_SIM/3510C_LVI = 1.0 µL</li> <li>5. 8270D_SIM_DOD5/3510C = 1.0 µL</li> <li>6. 8270D_SIM_DOD5/3510C_LVI = 1.0 µL</li> </ol> 1.0 µL injection uses Standard Method Calibration standards (Section 7.2.2) 5.0 µL injections uses LVI Method Calibration standards (Section 7.2.2)
Transfer Line	290 °C or 300 °C
<b>Mass Spectrometer Conditions<sup>1,2</sup></b>	
MS Source	230 °C or 240 °C
MS Quadrupole	200 °C
Dwell Time per Ion	Ranges from 30 to 100 milliseconds
Ions	See following tables

<sup>1</sup> The conditions listed above are subject to final fine adjustments to maximize instrument sensitivity. Changes to the above conditions are acceptable as long as method criteria are met.

<sup>2</sup> Details on the mass assignments in each window along with start and dwell times are given in Appendix III.

**Table II: Surrogates for Standard List Analysis**

<b>PAH Surrogates</b>	<b>Mass Ion</b>	<b>Confirmation Ion</b>
Nitrobenzene-d <sub>5</sub>	82	128
2-Fluorobiphenyl	172	171
Terphenyl-d <sub>14</sub>	244	122

**Table III: Internal Standards for Standard List Analysis**

<b>Compound</b>	<b>Mass Ion</b>	<b>Confirmation Ion</b>
Acenaphthene-d <sub>10</sub>	164	162
Phenanthrene-d <sub>10</sub>	188	94
Chrysene-d <sub>12</sub>	240	120

**Table IV: PAH Compounds and Ions Used for Analysis**

<b>Compound</b>	<b>Mass Ion</b>	<b>Confirmation Ion</b>
Acenaphthene	153	152
Acenaphthylene	152	151
Anthracene	178	179
Benzo(a)anthracene	228	226
Benzo(a)pyrene	252	253
Benzo(b)fluoranthene	252	253
Benzo(g,h,i)perylene	276	138
Benzo(k)fluoranthene	252	253
Chrysene	228	226
Dibenzo(a,h)anthracene	278	139
Dibenzofuran	168	139
Fluoranthene	202	101
Fluorene	166	165
Indeno(1,2,3,cd)pyrene	276	138
1-Methylnaphthalene	142	141
2-Methylnaphthalene	142	141
Naphthalene	128	129
Phenanthrene	178	179
Pyrene	202	101
Morpholine	57	87

**Table V: Example Retention Times, IS and Surrogate Associations**

<b>Compound</b>	<b>RT<sup>1</sup> (min.)</b>	<b>IS #</b>	<b>Surrogate #</b>
Morpholine	4.001	1	1
Naphthalene	5.921	1	1
2-Methylnaphthalene	6.595	1	1
1-Methylnaphthalene	6.700	1	1
Acenaphthylene	7.512	1	2
Acenaphthene	7.686	1	2
Dibenzofuran	7.861	1	2
Fluorene	8.210	1	2
Phenanthrene	9.194	2	2
Anthracene	9.255	2	2
Fluoranthene	10.768	2	2
Pyrene	11.166	2	2
Benzo(a)anthracene	13.827	3	3
Chrysene	13.924	3	3
Benzo(b)fluoranthene	17.004	3	3
Benzo(k)fluoranthene	17.089	3	3
Benzo(a)pyrene	18.034	3	3
Indeno(1,2,3,cd)pyrene	21.509	3	3
Dibenz(a,h)anthracene	21.583	3	3
Benzo(g,h,i)perylene	22.306	3	3
Acenaphthene-d <sub>10</sub> (IS)	7.657	1	-
Phenanthrene-d <sub>10</sub> (IS)	9.177	2	-
Chrysene-d <sub>12</sub> (IS)	13.856	3	-
Nitrobenzene-d <sub>5</sub> (Surr)	5.201	1	1
2-Fluorobiphenyl (Surr)	6.945	1	2
Terphenyl-d <sub>14</sub> (Surr)	11.38	2	3

<sup>1</sup>Retention times may vary depending upon chromatographic conditions.

**Table VI: DFTPP Key Ions and Ion Abundance Criteria  
 8270C**

<b>Mass</b>	<b>Ion Abundance Criteria</b>
51	30-60 % of mass 198
68	< 2 % of mass 69
69	Mass 69 relative abundance
70	< 2 % of mass 69
127	40-60 % of mass 198
197	< 1 % of mass 198
198	Base peak, 100 % relative abundance
199	5-9 % of mass ion 198
275	10-30 % of mass 198
365	> 1 % of mass 198
441	Present, but less than mass 443
442	40-100 % of mass 198
443	17-23 % of mass 442

With the exception of mass 442, the tune criteria for SW846 method 8270D are less stringent for the criteria required in SW846 method 8270C. For 8270D, the 442 mass must be greater than 50% of mass 198 to meet the tune criteria. By using the 8270C criteria, the rest of the data will be within the 8270D criteria.

**Table VII: 8270D Relative Response Factor Criteria for Initial and Continuing Calibration**

<b>Compound</b>	<b>Minimum RRF</b>	<b>Maximum %RSD</b>	<b>Maximum %Diff</b>
Acenaphthene	0.900	20	25
Acenaphthylene	0.900	20	25
Anthracene	0.700	20	25
Benzo(a)anthracene	0.800	20	25
Benzo(a)pyrene	0.700	20	25
Benzo(b)fluoranthene	0.700	20	25
Benzo(g,h,i)perylene	0.500	20	25
Benzo(k)fluoranthene	0.700	20	25
Chrysene	0.700	20	25
Dibenzo(a,h)anthracene	0.400	20	25
Dibenzofuran	0.800	20	25
Fluoranthene	0.600	20	25
Fluorene	0.900	20	25
Indeno(1,2,3,cd)pyrene	0.500	20	25
1-Methylnaphthalene	0.400	20	25
2-Methylnaphthalene	0.400	20	25
Naphthalene	0.700	20	25
Phenanthrene	0.700	20	25
Pyrene	0.600	20	25

**Table VIII**  
**Specific DoD QSM 5.0 and DOE QSAS 3.0 Requirements for 8270D**

This table includes components of the DoD QSM 5.0 and DoE QSAS 3.0 that are different from TestAmerica's standard procedures. For a complete description of the requirements, see DV-QA-024P. Also listed are the variances that TestAmerica is requesting for this analysis; these alternate criteria are only used with project-specific approval

Requirement	Variance (if allowed)	DoD QSM 5.0 and DoE QSAS 3.0
Initial Calibration Verification (ICV)	-- 4PP	All analytes must be within $\pm 20\%$ of the true value.  Allow $\pm 30\%$ of true value for known poor performers only if these compounds are not identified as critical compounds of concern by the client for the project under consideration.
Continuing calibration Verification (CCV)	-- 4PP 3HR 7MS	Run before sample and at the end of the analytical batch (end of 12 hours). Acceptance limits for all analytes is $\pm 20\%$ of true value for CCV at start of 12 hours.  Allow $\pm 30\%$ of true value for known poor performers only if these compounds are not identified as critical compounds of concern by the client  If the CCV is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project  Allow $\pm 50\%$ for end of analytical batch excluding poor performing compounds. Reanalysis performed due to failed closing CCV only for the analytes identified by the client as critical compounds of concern for the project, and to report qualified results for other analytes.  If any analytes fail in a CCV, recalibrate and re-analyze all affected samples or immediately (within one hour) analyze two consecutive CCVs and if both pass for the analytes that failed, the CCV is acceptable.
Internal Standards (IS)	-- 8ISRT	RT must be $\pm 10$ seconds from RT of the midpoint standard in the ICAL  RT must be $\pm 30$ seconds from RT of the midpoint standard in the ICAL. Daily routine column maintenance often results in larger RT changes than 10 sec. within a short time.
LCS	-- 4PP 3HR 1SME	Include all analyte(s) in LCS that are required to be reported, including surrogates, except those compounds listed as "Additional Analytes" by TestAmerica. These compounds are rarely requested and historical limits may not accurately reflect current performance.  If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project  Otherwise, correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample, then apply Q-flag to specific analyte(s) in all samples in the associated prep batch. Flagging is only appropriate when samples cannot be reanalyzed unless 3HR is accepted by the client.  Marginal exceedances are not allowed for critical chemicals of concern (risk drivers). Client must notify TestAmerica of these targets or if marginal exceedances will not be allowed.
Surrogates	-- 4PP	For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.  If surrogate recoveries are above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project.

## Appendix I: Extended List PAH Analysis by GC/MS

### Summary of Method

This is the extended list for the SIM analysis that some clients require. All of the compounds listed in this appendix are analyzed for in addition to the standard compounds discussed throughout this SOP.

### Modifications from the SIM analysis are as follows:

- The DFTPP tune has tailing factors that are calculated for Pentachlorophenol and Benzidine and a DDT breakdown check is performed.
- The instrument is calibrated at eight concentration levels. The calibration levels are made by diluting two stock standards with concentrations of 20 µg/mL [PAHXSIM stock (#1)] and 2µg/mL [PAHXSIM 2<sup>o</sup> stock (#2)] down to the concentrations listed below, in methylene chloride. All phthalate compounds and 2-methylnaphthalene are at a ratio of 2:1 in the stock standards. Therefore, if the concentration is 0.02 µg/mL for the target analytes, the phthalates are at 0.04 µg/mL.

Level (µg/mL)	Stock ID	Stock Amt (µL)	Solvent amount (µL)	IS amount (µL)	Final Volume (µL)
0.02 µg/mL	#2	5	495	50	500
0.1 µg/mL	#2	25	475	50	500
0.3 µg/mL	#2	75	425	50	500
0.6 µg/mL	#1	15	485	50	500
1.2 µg/mL	#1	30	470	50	500
2.5 µg/mL	#1	62.5	437.5	50	500
5.0 µg/mL	#1	125	375	50	500
10.0 µg/mL	#1	250	250	50	500

Response factors for each compound must be ≤ 20% RSD. If any compound is > 20% RSD, must use the best curve fit.

### Initial Calibration Verification

- The second source calibration stock is also at 20 µg/mL (PAHSIM SSV stock).
- The second source verification (SSV or ICV) is analyzed at 1.2 µg/mL.

- The acceptance criterion for the ICV is  $\pm 25\%D$ .

**Continuing Calibration Verification**

- The CCV is run at 0.6  $\mu\text{g/mL}$
- The criterion: The Average  $\%D$  for all compounds must be  $<20\%D$ , with no single compound exceeding  $30\%D$ .

**Sample extraction:** See DV-OP-0008 (aqueous) and DV-OP-0009 (soil).

**Sample concentration:** See DV-OP-0007.

**Sample analysis:**

- Internal Standard final concentration is 6  $\mu\text{g/mL}$  in standards and extracts. The stock is at 400  $\mu\text{g/mL}$
- For the MS/MSD, the recovery for the spike pair must be within the control limits stored in the LIMS system. The MS/MSD pair is generally aliquotted and run two times on the instrument, to confirm the results. If the results to be reported are from the first analysis, it is not required that the second analysis be within the 12 hour tune clock.

**Instrument Configuration:**

The GCMS instrumentation is configured the same as in the SIM analysis.

**Extended List Compounds, Reporting Limits and Ions Used for Analysis:**

Compound	Water Reporting Limit (ng/L)	Soil Reporting Limit ( $\mu\text{g/kg}$ )	Mass Ion	Confirmation Ion
1,4-Dioxane	NA	20	88	58
N-Nitrosodiphenylamine	1000	20	169	168
N-Nitrosodimethylamine	400	18	74	42
N-Nitrosodiethylamine (LVI)	100	--	102	44
N-Nitrosodi-n-propylamine (LVI)	100	--	70	42
Butyl Benzyl Phthalate	1000	20	149	91
Dimethyl Phthalate	1000	20	163	164
Diethyl Phthalate	1000	20	149	177
Bis(2-Ethylhexyl) Phthalate	1000	20	149	167
Di-n-octyl Phthalate	1000	20	149	150
Di-n-butyl Phthalate	1000	20	149	150

**Extended List Compounds Example Retention Times, IS and Surrogate Associations:**

Compound	RT <sup>1</sup> (min.)	IS #	Surrogate #
1,4-Dioxane	1.60	1	2
N-Nitrosodiphenylamine	6.75	2	2
N-Nitrosodimethylamine	2.16	1	2
N-Nitrosodiethylamine (LVI)	2.72	1	1
N-Nitrosodi-n-propylamine (LVI)	3.69	1	1
Butyl Benzyl Phthalate	10.33	2	2
Dimethyl Phthalate	5.92	1	2
Diethyl Phthalate	6.51	1	2
Bis(2-Ethylhexyl) Phthalate	11.67	2	2
Di-n-octyl Phthalate	13.69	3	2
Di-n-butyl Phthalate	7.95	2	2
Acenaphthene-d <sub>10</sub> (IS)	7.657	1	-
Phenanthrene-d <sub>10</sub> (IS)	9.177	2	-
Chrysene-d <sub>12</sub> (IS)	13.856	3	-
Nitrobenzene-d <sub>5</sub> (Surr)	5.201	1	1
2-Fluorobiphenyl (Surr)	6.945	1	2
Terphenyl-d <sub>14</sub> (Surr)	11.38	2	3

<sup>1</sup>Retention times may vary depending upon chromatographic conditions.

APPENDIX II

**Instrument Maintenance Schedules  
 Mass Spectrometer & Gas Chromatograph**

<b>MASS SPECTROMETER Instrument Maintenance Schedule</b>				
<b>Daily</b>	<b>Weekly</b>	<b>As Needed</b>	<b>Quarterly</b>	<b>Annually</b>
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43).	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check vacuum, relays, gas pressures, and flows.	Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.		Change the oil in the mechanical rough pump.
Check inlets, septa.		Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.		Relubricate the turbomolecular pump-bearing wick.
Check baseline level.		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates the need for replacement.		

**APPENDIX II (continued)**

**Instrument Maintenance Schedules  
 Mass Spectrometer & Gas Chromatograph**

<b>GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)</b>	
<b><i>Daily</i></b>	<b><i>As Needed</i></b>
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Check inlets, septa. Clean injector port.	Replace septa.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Repair or replace flow controller if constant gas flow cannot be maintained.
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

**APPENDIX III  
 Mass Spectrometer Settings for Single Ion Monitoring**

Group ID	Group Start Time <sup>1</sup> (min)	Analyte	Masses	Dwell Times
1	1.45	N-Nitrosodimethylamine	74, 42	50, 50
		1,4-Dioxane	88, 58	50, 50
		Morpholine <sup>2</sup>	57, 87	50, 50
		N-Nitrosodiethylamine (LVI) <sup>3</sup>	102, 44	50, 50
2	2.60	Nitrobenzene-d <sub>5</sub>	82, 128	50, 50
		Naphthalene	128, 129	50, 50
		N-Nitrosodiethylamine (LVI) <sup>3</sup>	102, 44	50, 50
		N-Nitrosodi-n-propylamine (LVI)	70, 42	50, 50
3	4.79	2-Fluorobiphenyl	172, 171	50, 50
		2-Methylnaphthalene	142, 141	50, 50
		1-Methylnaphthalene	142, 141	50, 50
4	5.46	Dimethyl Phthalate	163, 164	50, 50
		Acenaphthene- d <sub>5</sub>	164, 162	50, 50
		Acenaphthene	153, 152	50, 50
		Acenaphthylene	152, 151	50, 50
		Dibenzofuran <sup>4</sup>	168, 139	50, 50
5	6.06	Diethyl Phthalate	149, 177	50, 50
		N-Nitrosodiphenylamine	169, 168	50, 50
		Fluorene	166, 165	50, 50
		Dibenzofuran <sup>4</sup>	168, 139	50, 50
6	6.78	Phenanthrene-d <sub>10</sub>	188, 94	50, 50
		Phenanthrene	178, 179	50, 50
		Di-n-butyl Phthalate	149, 150	50, 50
		Anthracene	178, 179	50, 50
7	8.05	Butyl Benzyl Phthalate	149, 91	50, 50
		Terphenyl-d <sub>14</sub>	244, 122	50, 50
		Fluoranthene	202, 101	50, 50
		Pyrene	202, 101	50, 50
8	10.48	Chrysene- d <sub>12</sub>	240, 120	50, 100
		Bis(2-Ethylhexyl) Phthalate	149, 167	100, 100
		Chrysene	228, 226	50, 50
		Benzo(a)anthracene	228, 226	50, 50
9	12.33	Di-n-octyl Phthalate	149, 150	50, 50
		Benzo(a)pyrene	252, 253	50, 50
		Benzo(b)fluoranthene	252, 253	50, 50
		Benzo(k)fluoranthene	252, 253	50, 50
10	16.48	Dibenzo(a,h)anthracene	278, 139	50, 50
		Indeno(1,2,3-cd)pyrene	276, 138	50, 50
		Benzo(g,h,i)perylene	276, 138	50, 50

<sup>1</sup>Group start times may vary due to chromatographic conditions.

<sup>2</sup>Morpholine method detection limit verifications not kept current. Laboratory does not stock standards.

<sup>3</sup>N-Nitrosodiethylamine (LVI) elutes between windows 1 and 2 and was therefore included in both.

<sup>4</sup>Dibenzofuran elutes between windows 4 and 5 and was therefore included in both.



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## ICP Analysis for Trace Elements by SW-846 Method 6010C/D

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## **1.0 Scope and Application**

- 1.1** This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICPAES). This procedure references Methods 6010C and 6010D for hazardous waste (RCRA) testing.
- 1.2** The elements that can be determined by this procedure are listed in Attachment 1, together with the routine reporting limits. Additional elements may be analyzed under Method 6010C and 6010D provided that the method performance criteria presented in Section 12.0 are met.
- 1.3** The laboratory digests all water samples according to SOP DV-IP-0010.
- 1.4** Silver concentrations must be below 1.0 mg/L in aqueous sample digestates and 100 mg/kg in solid matrix sample digestates. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data. Samples with silver concentrations exceeding these levels must be re-prepared and reanalyzed using a smaller sample amount.
- 1.5** The digestion procedure for soil samples is described in SOP DV-IP-0015.
- 1.6** State or client specific requirements may take precedence over this SOP for water analyses. Review special instructions for each project before starting work.

## **2.0 Summary of Method**

- 2.1** The laboratory uses simultaneous ICPAES instruments, with both axial and radial viewing configurations. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs.
- 2.2** 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 a charge injection device (CID). The photo-currents from the charge injection device (CID) are processed and controlled by a computer system.
- 2.3** 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.
- 2.4** Refer to the appropriate SOPs for details on sample preparation methods: DV-IP-0010 for aqueous samples and DV-IP-0015 for soil samples.

### 3.0 **Definitions**

- 3.1 **Dual View ICP** – an ICP equipped with both radial and axial viewing capabilities.
- 3.2 **Dissolved Metals** - Those elements which pass through a 0.45- $\mu$ m membrane. The sample is acidified after filtration.
- 3.3 **Potentially Dissolved Metals** - Potentially dissolved metals is the concentration of metals in solution after acidifying the sample with nitric acid to pH <2, holding at room temperature for 8 to 96 hours, and then filtering through a 0.45- $\mu$ m membrane filter. This definition is based on the Colorado surface water regulations.
- 3.4 **Suspended Metals** - Those elements which are retained by a 0.45- $\mu$ m membrane.
- 3.5 **Total Metals** - The concentration determined on an unfiltered sample following vigorous digestion.
- 3.6 **Total Recoverable Metals** - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.
- 3.7 **Reporting Limit (RL)** - The lowest concentration to which results are reported without qualification. Details concerning RLs are presented in Policy DV-QA-009P.
- 3.8 **Reagent Water** - Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.
- 3.9 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

### 4.0 **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 the following:
- 4.1.1 Overlap of a spectral line from another element.
  - 4.1.2 Unresolved overlap of molecular band spectra.
  - 4.1.3 Background contribution from continuous or recombination phenomena.
  - 4.1.4 Stray light from the line emission of high concentration elements.
- 4.2 A background correction technique is used 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.

#### **4.3 Spectral Interferences**

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 signal. 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 must be applied to the analyte to compensate for the effects of these unwanted emissions.

#### **4.4 Physical Interferences**

An internal standard (IS), yttrium or other suitable element, is added to all solutions to correct and monitor physical interferences. Use of a peristaltic pump and the mass flow controller also help to overcome physical interferences. 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 internal standard recoveries are not acceptable (see Section 9.11), then dilution of the sample may be necessary to overcome the interferences.

#### **4.5 Chemical Interferences**

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not significant with the ICP technique, but if observed, can be minimized by buffering the sample, matrix matching, or standard addition procedures.

### **5.0 Safety**

**5.1** Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

**5.2** 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, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

#### **5.3 Specific Safety Concerns or Requirements**

**5.3.1** Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be

removed and discarded; non-disposable gloves must be cleaned immediately.

**5.3.2** The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

**5.4 Primary Materials Used**

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

<b>Material <sup>(1)</sup></b>	<b>Hazards</b>	<b>Exposure Limit<sup>(2)</sup></b>	<b>Signs and Symptoms of Exposure</b>
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.			

**6.0 Equipment and Supplies**

**6.1 Instrumentation**

**6.1.1** Thermo Fischer ICP 6500E Trace Analyzers are currently used.

Instruments with demonstrated equivalent performance can also be used

- 6.1.2 Radio Frequency Generator
- 6.1.3 Argon gas supply
- 6.1.4 Coolflow or appropriate water-cooling device.
- 6.1.5 Peristaltic Pump.
- 6.1.6 Autosampler.

## 6.2 Supplies

- 6.2.1 Calibrated automatic pipettes or Class A glass volumetric pipettes.
- 6.2.2 Class A volumetric flasks.
- 6.2.3 Autosampler tubes.

## 6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

## 7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All standards used in calculations shall be entered into the TALS Reagent Module with all applicable information (e.g., components, concentrations, expiration, etc.).

### 7.2 Shelf-Life

- 7.2.1 Stock standards, standards as received from the vendor, expire on the date assigned by the vendor. If no date is assigned by the vendor, a one-year expiration will be assigned by the laboratory.
- 7.2.2 The expiration date of intermediate concentration standards or working standards will be set at six months or less and cannot be later than the date assigned to any of the stock standards used to prepare the intermediate solution.
- 7.2.3 If visible deterioration is noted for any standard, it must be re-verified against a second-source. Any standard that does not verify must be replaced immediately.

### 7.3 Standards

**7.3.1** Standards used for calibration and quality control purposes must be NIST traceable, where available. Multi-component custom blend standards must be verified against a second-source standard before they are first put into use (the only exception is standards purchased directly from NIST), as described in SOP DV-QA-0015. If the standard has been purchased previously it does not need to be verified, but the COA must be inspected to confirm that there have been no changes to the standard analyte levels.

**7.3.2** Stock standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon, polyethylene, or polypropylene bottles. Silver standards must be protected from light. The preparation frequency is governed by the parent standard with the earliest expiration date unless specified otherwise in this SOP. Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Reagent Module in the TestAmerica LIMS TALS.

**7.3.3** Intermediate calibration and QC standards are prepared in water with hydrochloric and nitric acids in order to approximate the acidic matrix of the various digests analyzed. This is an important point. Even with the use of yttrium as an internal standard, deviations from these concentrations can cause physical effects, as discussed in Section 4.4 of this procedure.

### 7.4 Reagent Blank / Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

Fill a 20-liter carboy with about 18 liters of reagent water. Slowly add 1 liter of concentrated nitric acid and 1 liter of concentrated hydrochloric acid. Adjust the total volume to 20 liters. Mix carefully. Record the acid lot number and other required information in the Blank Reagent Logbook stored in the metals prep area.

### 7.5 Stock ICSA and ICSAB Standards

The following standards are purchased from commercial sources:

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
lcp stk ICSA	Fe Al, Ca, Mg	2,000 5,000
ANALYTES B	Ba, Be, Co, Cr, Cu, Mn, V Ag, Cd, Ni, Pb, Zn	50 100
ICP ISAB STD1	Li, Mo, Sb, Sr As, B, P Se	100 200 500

Stock ICSA & ICSAB Standard	Elements	Concentration (mg/L)
	K, Na	5,000
ICP ISAB STD2	Ti Sn	100 1,000
10000 Si	Si	10,000
10000 Th	Th	10,000
1000 TI	Tl	1,000
1000 Zr	Zr	1,000
1000 S	S	1,000
1000 Bi	Bi	1,000

#### 7.6 ICSA Working Standard (ICP ICSA)

A combined working ICSA standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSA and ICSAB Standards:

Stock Standard	Volume of Stock Added (mL)
ICSA Std	25

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSA standard concentrations shown in Attachment 4.

#### 7.7 ICSAB Working Standard

A combined working ICSAB standard is made in a 250-mL volumetric flask using the following volumes of the Stock ICSAB Standards:

Stock Standard	Volume of Stock Added (mL)
Icp stk ICSA	25
ANALYTES B	2.5
ICP ISAB STD1	2.5
ICP ISAB STD2	2.5
10000 Si	0.25
10000 Th	0.05
1000 TI	2.5
1000 Zr	0.25
1000 S	0.25
1000 Bi	0.25

Adjust to volume (250 mL) using the reagent blank solution. This produces the final ICSAB standard concentrations shown in Attachment 4.

**7.8 Calibration Check Standard (S1, S2)**

The two calibration check standards are the same as the working ICAL standards (ICP ICAL1A and ICP ICAL2A) described in Section 7.12.

**7.9 Laboratory Control Sample (LCS) Stock Standards**

The LCS stock standards are purchased from commercial sources. The stocks are custom-made standards purchased at ready-to-use concentrations as follows:

<b>LCS Stock Standards</b>	<b>Elements</b>	<b>Concentration (mg/L)</b>
ICP SPK 3A	Ca, K, Mg, Na	5,000
	P	1,000
	Al, Ba, Bi, Se, Tl, U,	200
	As, Fe, Li, Sr, Th	100
	Co, Mn, Ni, Pb, V, Zn	50
	Cu	25
	Cr	20
	Cd	10
	Ag, Be	5
	ICP SPK 2B	Sb, Zr
B, Mo, Ti		100
Sn		200
Si		1,000
(SiO <sub>2</sub> )		(2,140)
S		200

The soil and water LCSs are prepared according to the instructions in SOPs DV-IP-10 and DV-IP-0015. Final concentrations are shown in Attachment 2.

**7.10 Matrix Spike / Matrix Spike Duplicate (MS/MSD)**

The same LCS stock standards described in Section 7.9 are also used to prepare matrix spikes and matrix spike duplicates. Final concentrations are shown in Attachment 2.

**7.11 Post Digestion Spike (PDS) Standards (Analyte Addition Spike Standards)**

The custom standards tabulated below are purchased from a commercial source. Add 0.06 mL of each to 6 mL (100X) of digestate or dilution of digestate.

PDS Stock	Elements	Conc. (mg/L)
ICP PDS 1	Ag, Be, Cd, Co, Cr, Cu,	5
	Mn, Ni, Sr, V	5
	Ba, Li, Pb,	10
	As, Se, Th, Tl, Zn	20
	U	50
	Al, Fe	100
	P	200
	Ca, K, Mg, Na,	2,000
	ICP PDS 2	Mo, Ti, Zr
	B, Sb, Sn	10
	Si	500
	(SiO <sub>2</sub> )	(1,070)

## 7.12 Initial Calibration (ICAL) Standards

### 7.12.1 Stock Calibration Standards

The following stock solutions are purchased from commercial sources.

Stock Standard	Elements	Conc. (mg/L)
Icp cal std 2	Mo, Ti, Zr	100
	Sn	200
	Si	1,000
	(SiO <sub>2</sub> )	(2,140)
Icp cal std 3	Ag, Al, B, Ba, Be, Cd, Co, Cr, Cu, Mn,	100
	Ni, Sr, V, Zn	100
	Li, P	200
	Fe	500
	Ca, Na	1,000
	Mg	4,000
	K	10,000
	Al, Ca, Fe, Na, S, Th Stocks	Al, Ca, Fe, Na, S, Th
As, Pb, Sb, Se, Tl, U, Bi Stocks	As, Pb, Sb, Se, Tl, U, Bi	1,000

### 7.12.2 Working Initial Calibration Standard (ICP ICAL1A)

Add 5.0 mL each of Icp cal std 2 and Icp cal std 3 to a 500-mL volumetric flask partially filled with reagent blank solution. Add 1 mL of the As, Pb, Sb, Se, and Tl stocks. Dilute to the mark with reagent blank solution.

### 7.12.3 Working Initial Calibration Standard (ICP ICAL2A)

Add 10 mL of the Al and Fe and 50 mL of the Na 10,000 mg/L stock solutions; 1 mL of the Th and 20 mL of the U 1,000 mg/L stock solutions; 2 mL of the 1,000 mg/L Bi solution and 1 mL of the 10,000 mg/L S solution to

a 1,000-mL volumetric flask partially filled with reagent blank and dilute to the mark with reagent blank.

### 7.13 Initial Calibration Verification (ICV)

#### 7.13.1 ICV Stock Standards

The following stock solutions are purchased from commercial sources:

Stock Standard	Elements	Conc. (mg/L)
Icp ICVL A	Al, As, B, Ba, Be, Cd, Co, Cr, Cu, Fe, Li, Mn,	25
	Ni, Pb, Sr, V, Zn	25
	Se, Tl	50
	Ca, Na	200
	Mg	1,000
	K	2,000
Icp ICVL B	Ag, Mo, Sb, Ti, Zr	25
	Sn	50
	P, Si	200
	(SiO <sub>2</sub> )	(428)
Icp ICVH	Al, Na	4,000
	Fe	8,000
	U	500
	Th	300
Bi, S Stocks	Bi, S	1,000

#### 7.13.2 Working High Initial Calibration Verification (ICP ICVH)

Add 1.0 mL of the ICVH Stock, 0.05 ml Bi and 0.4 mL of the Sulfur to a 100 mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

**Note:** For Method 6010D the ICV working solutions must be prepared daily.

#### 7.13.3 Working Initial Calibration Verification (ICP ICV)

Add 1.0 mL of each of the Icp ICVL A and Icp ICVL B stock solutions to a 100-mL volumetric flask partially filled with reagent blank solution and dilute to the mark.

**Note:** For Method 6010D the ICV working solutions must be prepared daily.

**7.14 Reporting Limit Standard (RLSTD)**

**7.14.1 RL Stock Standard**

The following stock solutions are purchased from commercial sources:

Standard	Elements	Conc. (mg/L)
ICP RLSTD 1A	As, Sb, Se, Tl	10
	Pb	3
ICP RL STD 2A	Mo, Ti, Zr	10
	Sn	20
	Si	500
	(SiO <sub>2</sub> )	1,070
ICP RL STD3A	Ag, Cr, Cu, Li, Ni, Th, V, Zn,	10
	Al, B	100
	Ba, Cd, Co, Sr	5
	Be	1
	Ca, Mg	200
	Fe	30
	K, Na, P	1,000
	Mn	3
	U	60
	100 mg/L S	S
100 mg/L Bi	Bi	100

**7.14.2 Daily Reporting Limit Standard (ICP CRI)**

Add 0.1 mL of each of ICP RLSTD 1A, ICP RL STD 2A, ICP RL STD 2A, 100 mg/L Bi and 100 mg/L S to a 100-mL volumetric flask partially filled with reagent blank and dilute to the mark. The Working RL standard must be prepared fresh each day.

**7.15 High Continuing Calibration Verification (ICP CCVH)**

Perform a 2x dilution of the working ICP ICAL2A solution (Section 7.12.3) with reagent blank solution.

**7.16 Continuing Calibration Verification (ICP CCV)**

Perform a 2x dilution of the working ICP ICAL1A solution (Section 7.12.2) with reagent blank solution.

**7.17 Low Level ICV/Low Level CCV (ICP LLCCV)**

The low level ICV/CCV verification stock standards are custom-made commercial standards as follows:

LLICV/LLCCV Stock Standard	Elements	Conc. (mg/L)
ICP LLCCV-1	K	300
	Na	100
	Ca, Mg	20
	Al, Bi, Fe	10
	U	6
	Ni	4
	Zn	2
	As, Cu, Se, Tl, Th	1.5
	Ba, Cr, Co, Li, Mn, Ag, Sr,	1
	V	1
	Pb	0.9
	Cd	0.5
	Be	0.1
ICP LLCCV-2	P	300
	Si	50
	B	10
	Sn	10
	Mo	2
	Zr	1.5
	Sb	1
	Ti	1

#### 7.17.1 Low Level ICV \ Low Level CCV, Working Standards

RL Standard	Vol. of Stock Added (mL)
ICP-LLCCV-1	1
ICP-LLCCV-2	1

Adjust to volume (100 mL) using the reagent blank solution.

#### 7.18 Linear Range Verification Standard (LR)

The LRA standard is prepared from single element stock standards of each metal obtained from a commercial source. The stock standards are each purchased at a concentration of 1,000 mg/L except for Iron and Silicon which are each at a concentration of 10,000 mg/L. The LRA is prepared by taking the appropriate volume of each stock and adding it to a 500 mL volumetric flask partially filled with reagent blank and diluted to the mark after all elements have been added. The volume of each stock solution of each metal and volume used, along with final concentrations of each are listed in the following table.

Elements	Stock Conc. (mg/L)	Volume of stock (mL)	Final Conc. (mg/L)
Cd,	1,000	1.0	2
Co, Mo, Se, Tl	1,000	2.5	5
As, B, Cr, Cu, Mn, Ni, Pb, Sr, Ti, V, Zn	1,000	5.0	10
Ba	1,000	6.0	12
Fe	10,000	25	500
Si (SiO <sub>2</sub> )	10,000	2.5	50 (107)

### 7.19 Reagents

7.19.1 Concentrated nitric acid (HNO<sub>3</sub>), trace metals grade or better.

7.19.2 Concentrated hydrochloric acid (HCl), trace metals grade or better.

7.19.3 Reagent water must be produced by a Millipore DI system or equivalent, with a minimum resistivity of 1.0 Mohm/cm at 25 °C.

### 8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation <sup>1</sup>	Holding Time <sup>2</sup>	Reference
Waters	HDPE	50 mLs	HNO <sub>3</sub> , pH < 2;	180 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool ≤ 6 °C <sup>3</sup>	180 Days	N/A

<sup>1</sup> 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 for most programs. Preservation must be verified prior to analysis.

<sup>2</sup> Inclusive of digestion and analysis.

<sup>3</sup> Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot volume for both analyses must be refrigerated.

### 9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply.

- 9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.
- 9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. DoD QSM 5.0 QC Acceptance Criteria for ICP analyses are presented in Attachment 11. The criteria must be met unless otherwise documented in the project documents.
- 9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.
- 9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

## 9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See Policy DV-QA-003P for further details.

## 9.3 Method Blank

The blank is de-ionized water taken through the procedure as if it were a sample. For soil samples analyzed under the DoD QAPPs, the method blank consists of < 1 mm glass beads that have been processed in the same manner as the samples. A method blank is required with every batch of 20 or less samples.

**Acceptance Criteria:** The method blank must not contain any analyte of interest above  $\frac{1}{2}$  the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

**Corrective Action:** If the method blank exceeds allowable levels, all associated samples must be redigested and reanalyzed. A possible exception is the situation in which the analyte is not detected in any of the associated samples, but this can only be done with client approval and it must be addressed in the final report case narrative.

#### 9.4 Laboratory Control Sample (LCS)

The LCS is prepared as described in Section 7.9. One LCS is required with each analytical batch.

**Acceptance Criteria:** The recovery of the LCS must be within historical control limits. Historical control limits are based on three standard deviations of past results, and must be 80 - 120% or tighter. In the instance where the LCS recovery is greater than 120% 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. The process of establishing control limits is described in more detail in the Policy DV-QA-003P. The control limits are stored in TALS.

**Corrective Action:** If the LCS recovery falls outside of the established limits, all associated samples must be redigested and reanalyzed

#### 9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS/MSDs are prepared as described in Section 7.10. One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require the MS/MSDs to be run at a 10% frequency. Some client specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing on only the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Note that if client instructions on the chain of custody form tell the lab to use a field blank for the MS/MSD, this should be double-checked with the laboratory PM.

**Acceptance Criteria:** The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at  $\pm 3$  standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

**NOTE:** DoD QSM 5 limits apply to projects performed under this program.

**Corrective Actions:** The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The

following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as "NC" (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as "NC" (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

**NOTE:** See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

**NOTE:** Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

**NOTE:** This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, an LCS and LCSD will be used to measure precision.

## 9.6 Method of Standard Additions (MSA)

**9.6.1** This technique involves constructing a calibration curve in the sample matrix itself to compensate for any sample interferences 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 that cause a baseline shift. Attachment 8 provides more guidance on performing MSA analyses.

**9.6.2** EPA Method 1311 (Section 8.4) requires that the MSA be used as the calibration method if the MS or MSD recoveries for TCLP extracts are less than 50% and the sample result is within 80 - 100% of the regulatory level. Attachment 4 provides a list of the regulatory limits. Although the MSA calibration technique may be used with only the sample and a single spiked point, Method 1311 specifies that three spiked points must be used along with the sample.

**9.6.3** TALS does not currently have the capability to report results from an MSA-based analysis. If an MSA must be performed, the sample results must be calculated using the MSA spreadsheet (stored at R:\QA\Edit\Forms\Metals\MSA Worksheet - Water) and reported in an NCM. All of the associated samples must then be recalculated against the MSA spreadsheet. The completed spreadsheet must be saved and attached to the analytical batch in TALS along with the raw data.

**9.6.4** A manual "N" flag must also be added to all of the affected samples in TALS, indicating the presumptive evidence for the analyte. This flag signals the Project Manager and indicates that narration of the result is required.

## 9.7 Serial Dilution Test

A dilution test is performed for each batch of samples. The purpose of this test is to ensure that neither positive nor negative interferences are biasing the analytical results. The serial dilution test should be performed on the same sample used to perform the MS/MSD.

**Acceptance Criteria:** If the analyte concentration is sufficiently high (minimally, a factor of 50 times the MDL), an analysis of a 1:5 dilution (e.g., 1 mL of sample diluted to 5 mL with reagent blank solution) must agree within  $\pm 10\%$  of the original determination. For DoD QSM 5.0 the serial dilution is required if the MS or MSD fails and the parent concentration is greater than 50x the LOQ prior to dilution. 6010D requires the parent sample to be at least 25x higher than the RL to be calculable and sets the recovery limit at 20%.

**Corrective Action:** If the two results do not agree within the required limits, then a chemical or physical interference is suspected. A qualifier flag is assigned to the data and the failure is addressed in the case narrative to alert the client that a matrix affect may be present. For DoD QSM 5.0 a J-flag is added to the parent sample for the specific analyte if the acceptance criteria are not met.

## 9.8 Post Digestion Spike (PDS)

Whenever the MS/MSD recoveries are unacceptable, a PDS spike must be performed. The PDS spike is prepared as described in Section 7.10. Some programs (e.g., AFCEE) require a PDS analysis whenever the serial dilution test fails. Other programs (e.g., DoD QSM 5.0) require a PDS to be included in every batch. Check project requirements. For programs where a PDS is required, the same sample that was used for the serial dilution test should be used for the PDS.

**Acceptance Criteria:** An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 80 - 120% for Method 6010C and 75 - 125% for 6010D.

**Corrective Action:** If the spike is not recovered within the specified limits, a matrix effect is confirmed. For DoD QSM 5.0 a J-flag is added to the parent sample if the sample concentration is less than 50x the LOQ prior to dilution. Any failures are flagged and should be described in the report case narrative.

## 9.9 Interference Check Analysis (ICSA / ICSAB)

The ICSA contains only interfering elements, the ICSAB contains analytes and interferences. Refer to Sections 7.5, 7.6, and 7.7 for the preparation of the ICSA and ICSAB solutions. Attachment 4 lists the final concentrations. All analytes are spiked

into the ICSAB solution. The ICSA and ICSAB solutions are analyzed at the beginning of the run.

**Acceptance Criteria:** The ICSAB results for the all analytes must fall within 80-120% of the true value. If any ICSAB analyte result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the samples rerun.

The absolute value of ICSA results for the non-interfering elements must be  $\leq 2 \times \text{RL}$ . The DoD and AFCEE programs have their own criteria based on the version used. For DoD QSM 5.0 the non-spiked analytes must be less than the absolute value of the LOD unless they are verified impurities. For 6010D the non-spiked analytes must be less than the absolute value of the RL.

**Corrective action:** If the ICSA results for the non-interfering elements do not meet these limits, 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. The sample data may also be accepted if the affected element was not required. If the interfering elements are not present in the field sample at a concentration which would result in an absolute value  $> 2x \text{RL}$ , 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  $> 2x \text{RL}$ , 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 do not meet the above conditions, then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed.

#### 9.10 Monitoring Internal Standard Results

Yttrium is automatically added as an internal standard (IS) to every solution tested through use of a third pump channel and mixing coil. 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).

**Acceptance Criteria:** If the internal standard counts fall within  $\pm 30\%$  of the counts observed in the ICAL blank, then the data are acceptable.

**Corrective Action:** If the internal standard counts in the field samples are outside of the control limits, the field samples must be diluted and reanalyzed;

## **10.0 Procedure**

**10.1** One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

**10.2** Any unauthorized deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

### **10.3 Sample Preparation**

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-IP-0010 and DV-IP-0015).

### **10.4 Calibration**

#### **10.4.1 Instrument Start Up**

Set up the instrument with the operating parameters recommended by the manufacturer. Complete any required preventative maintenance and record in the ICPAES Preventative Maintenance Log. Preventive maintenance recommendations are listed in the TestAmerica Denver Quality Assurance Manual. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

#### **10.4.2 Initial Calibration (ICAL)**

The calibration curve is established on each day of operation using a blank and one standard. The preparation of the ICAL standards is described in Section 7. The final concentrations of the ICAL standards are presented in Attachment 6. The validity of the calibration curve is confirmed by analysis of the ICV, CCV, ICB, RL Check standard and Low Level ICV/CCV) which are run immediately after the ICAL. Some programs also require a high-level verification check (see Section 10.4.9).

#### **10.4.3 Initial Calibration Verification (ICP ICVH and ICP ICV)**

Calibration accuracy is verified using a second-source standard (ICP ICVH and ICP ICV) that is at or below a concentration near the mid-point of the working range. The ICV is analyzed immediately after the ICAL. The preparation of this standard is described in Section 7. The concentration of the ICV standard is presented in Attachment 6.

**Acceptance Criteria:** The ICV result must fall within 10% of the true value for that solution. The relative standard

deviation must be < 5% (the laboratory is using at least two exposures for all ICP analyses).

**Corrective Action:** If the ICV fails to meet acceptance limits, the standard may be reanalyzed without modification to the instrument operating conditions. Two consecutive, acceptable analyses are required before the analytical run may continue. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

#### 10.4.4 Mid Level Continuing Calibration Verification (CCV)

The preparation of the CCV solution is described in Section 7. The final concentrations of the CCVs are presented in Attachment 6. Note that the CCV is made at a different concentration than the ICV to meet NELAC requirements. CCVs are analyzed after the ICV, after every ten samples, and at the end of the analytical run.

**Acceptance Criteria:** The CCV must be within 10% of the expected value. The relative standard deviation must be <5%.

**Corrective Action:** If the CCV fails to meet any of these criteria, the standard may be reanalyzed without modification to the instrument operating conditions. Two consecutive, acceptable analyses are required before the analytical run may continue. Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed.

#### 10.4.5 6010C - Low Level Initial Calibration (LLICV) and Continuing Calibration Verification (LLCCV)

The preparation of the LLCCV solution is described in Section 7. The low-level CCV needs to be analyzed at the beginning and end of every run sequence. If low level samples are expected then the low-level CCV should also be run every ten samples.

**Acceptance Criteria:** The LLCCV must be within +/-30% of the expected value to meet Method 6010C requirements.

**Corrective Action:** If the LLCCV fails to meet any of these criteria, the standard may be reanalyzed without modification to the instrument operating conditions. Two consecutive, acceptable analyses are required before the analytical run may continue. If the calibration cannot be verified within these specified limits, the analysis of samples containing the affected analytes at similar concentrations

cannot continue until the cause is determined and the LLCCV standard successfully analyzed. Otherwise, the instrument must be recalibrated and the samples reanalyzed since the last successful CCV must be reanalyzed. TestAmerica will not hold samples with concentrations greater than 10x the reporting limit to the 30% acceptance criteria.

#### 10.4.6 Initial Calibration Blank (ICB)

System cleanliness is verified by analyzing an ICB after the first CCV. The preparation of the ICB is described in Section 7.

**Acceptance Criteria:** Absolute values for the calibration blanks must be less than  $\frac{1}{2}$  the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD QSM 5.0 requires control of blanks to a concentration less than or equal to the LOD.

**Corrective Action:** If the ICB fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

#### 10.4.7 RL Calibration Check Standard (ICP CRI)

Calibration accuracy at the RL is verified by analyzing a standard prepared at a concentration at or below the laboratory's standard reporting limit. The preparation of this standard is described in Section 7. Alternate RLSTD concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Reagents Module in TALS.

**Acceptance Criteria:** For routine work the acceptance limits are  $\pm 50\%$  of the expected value. For **6010D and DoD QSM** the acceptance limits are  $\pm 20\%$ .

**Corrective Action:** If the RL Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

#### 10.4.8 Lower Limit of Quantitation Check (LLQC)

The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits, quarterly and on an

as needed basis to demonstrate the desired detection capability. The difference between the LLQC and the LLICV/CCV is that this standard is carried through the entire preparation and analytical procedure. Prepare 7 aliquots spiked at the LLOQ.

**Acceptance Criteria:** LLQC is verified when all analytes are detected within  $\pm 30\%$  of their true value with an RSD  $\leq 20\%$ .

**Corrective Action:** If the LLQC fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

#### 10.4.9 High-Level Calibration Check Standard

The method 6010C defines the linear working range used for daily analysis based on the LDR studies performed every six months, in which case this standard is not required. However, some programs require verification of the high end of the linear range at different frequencies. The DoD QSM 5.0 requires that the linear range must be verified on a daily basis. For DoD QSM 5.0 and Method 6010D samples, the spike level of the highest standard analyzed defines the linear range for that day.

**Acceptance Criteria:** The result for this standard must be within 10% of the expected value.

**Corrective Action:** If the High-Level Calibration Check standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analyst must run a standard at a lower concentration until the criteria is met for this calibration or the sample results cannot exceed the level of the highest calibration standard.

#### 10.4.10 Continuing Calibration Blank (CCB)

CCBs, prepared as in Section 7.4, are analyzed after each CCV.

**Acceptance Criteria:** Absolute values for the calibration blanks must be less than  $\frac{1}{2}$  the standard RL. Common lab contaminants such as sodium must be less than the RL. Client specific requirements take precedence. For example, DoD requires control of blanks to a concentration less than or equal to the LOD. Method 6010D sets the CCB upper limit at the RL.

**Corrective Action:** If the CCB is greater than these limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, instrument maintenance should be considered, the calibration re-verified, and all samples analyzed since the last successful CCB must be reanalyzed.

## 10.5 Sample Analysis

### 10.5.1 Replicate Readings

The laboratory averages the results from two exposures for Axial and Dual View ICP for each standard, field sample, and QC sample due to sample volume limitations of the autosampler tube.

### 10.5.2 Rinse Time between Samples

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 12.7, it can be demonstrated that a shorter rinse time may be used.

### 10.5.3 The following analytical sequence is used:

- Instrument Calibration
- High Standard Verification
- ICV
- LLICV (6010C only)
- CCV
- ICB
- RL Verification Standard
- LLQC (as needed)
- ICSA
- ICSAB
- LRA
- CCV
- CCB
- LLCCV (6010C only)
- 10 samples
- CCV
- CCB
- LLCCV (6010C)
- 10 samples
- CCV
- CCB
- LLCCV (6010C)
- Repeat sequence with 10 samples between CCV/CCB pairs
- CCV
- CCB
- LLCCV (6010C)

**10.5.4** Full method-required QC must be available for each wavelength used in determining reported analyte results. Guidelines are provided in the appendices for minimizing contamination of samples and standards (Attachment 10) and troubleshooting (Attachment 9).

**10.5.5 Dilutions for High Levels of Elements of Interest**

For 6010, results must fall within the linear range. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. Dilutions must be prepared using the reagent blank solution to maintain the correct acid concentration.

**10.5.6 6010D Mid-Run Recalibration**

During the course of an analytical run, the instrument may be recalibrated to correct for instrument drift. A recalibration must then be followed immediately by a new analysis of a CCV and CCB before any further samples may be analyzed.

**10.5.7 Dilutions for High Levels of Interfering Elements**

Dilutions are also required for an element that is included in an IEC calculation if it exceeds the linear range. If a dilution is not performed, the IEC may be inaccurately applied. Therefore, even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted until the interferent is at or below the working range. An NCM will be written in these instances.

**10.6 Instrument Maintenance**

See Section 20 in the QAM.

**10.7 Troubleshooting**

See Attachment 9.

**11.0 Calculations / Data Reduction**

**11.1** Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves & Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

**11.2** The procedure for performing the calculation of ferric iron is detailed in the Work Instruction WI-DV-0092, *Calculation Methods*.

**11.3** ICV percent recoveries are calculated according to the following equation:

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

**11.4** CCV percent recoveries are calculated according to the following equation:

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

**11.5** Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left( \frac{SSR - SR}{SA} \right) \times 100\%$$

Where:

SSR = Spike Sample Result  
 SR = Sample Result  
 SA = Spike Added

The relative percent difference (RPD) of a matrix spike/matrix spike duplicate pair is calculated according to the following equation:

$$RPD = \left[ \frac{|MSD - MS|}{\left( \frac{MSD + MS}{2} \right)} \right] \times 100$$

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:

$$\text{Final Concentration (mg/L)} = \frac{C \times V1 \times D}{V2}$$

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:

$$\text{Final Concentration (mg/kg), dry weight} = \frac{C \times V \times D}{W \times S}$$

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 LCS percent recovery is calculated according to the following equation:

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

**11.9** The IEC’s are calculated according to the following equation:

$$IEC = \left( \frac{\text{observed concentration}}{\text{observed concentration of the interfering element}} \right)$$

**11.10** The dilution test percent difference for each component is calculated as follows:

$$\% \text{ Difference} = \frac{|I - S|}{I} \times 100$$

Where:

I = Sample result (Instrument reading)

S = Dilution test result (Instrument reading × 5)

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

### **11.11 Documentation and Record Management**

**11.11.1** All sample data is uploaded to TALS. All sample preparation and analytical batch information, including the batch number(s), list of samples, preparation analyst and date, instrument analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes) is recorded in TALS.

**11.11.2** Raw data is scanned or saved directly as a PDF and is attached to the analytical batch in TALS.

**11.11.3** The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on a Data Review Checklist. See SOP DV-QA-0020 for more detail on the review process. Any manually transcribed data must be reviewed in its entirety by the second level data reviewer.

**11.11.4** If dilutions are required due to insufficient sample, interferences, or other problems, the reporting limit and MDL are multiplied by the dilution factor and the data may require flagging.

**NOTE:** Unless special instructions indicate otherwise, sample results less than the reporting limit are reported as ND.

## **12.0 Method Performance**

### **12.1 Method Detection Limit Study (MDL)**

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy DV-QA-005P. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD and DOE projects, an MDL verification is performed quarterly. DoD QSM 5.0 requires the MDLV spike level to be 2 - 4 times the calculated MDL.

### **12.2 Instrument Detection Limit Study**

**12.2.1** Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each wavelength used for analysis.

**12.2.2** Run seven blanks on three non-consecutive days.

**12.2.3** Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

**12.2.4** See Policy DV-QA-014P for a discussion of IDL studies and evaluation of IDL results.

**12.2.5** For Method 6010D the IDL solutions:

- Should be prepared with each of the different matrices analyzed on the instrument;
- Should be prepared with 10 replicates for each matrix;
- Should have all the replicates for each matrix analyzed in a single day.

### **12.3 Linear Dynamic Range (LDR)**

**12.3.1** The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample.

**12.3.2** The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

**12.3.3** The LDR is determined by analyzing successively higher standard concentrations of the analyte. A minimum of three standards is required for the initial and on-going studies, and one of the levels must be close to the

upper end of the range. The highest concentration must be within 10% of the stated concentration.

- 12.3.4 The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions. Certain programs do not allow the use of LDRs for reporting purposes and instead require all sample results to fall below the highest daily standard analyzed.
- 12.3.5 If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

#### **12.4 Background Correction Points**

- 12.4.1 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 of interest and record the apparent emission intensity from all other method analytes. 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.
- 12.4.2 Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the ICP instrument manual for specific procedures to be used in setting background correction points.

#### **12.5 Interelement Corrections (IECs)**

- 12.5.1 ICP interelement correction (IEC) 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 re-determined.
- 12.5.2 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.
- 12.5.3 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 produces a false analytical result with an absolute value greater than the RLs shown in Attachment 1. Note that the USACE program requires a control limit of  $2x$  [MDL], which is feasible when verified MDLs are used.

- 12.5.4 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." Method 6010D requires that the IEC standards include Al, B, Ba, Ca, Cu, Fe, Mg, Mn, Mo, Na, Ni, Se, Si, Sn, V, and Zn.
- 12.5.5 Dual-View 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 CID detector instruments as reflected by the ICSA response.

## 12.6 Rinse Time Determination

- 12.6.1 Rinse times must be determined annually.
- 12.6.2 To determine the appropriate rinse time for a particular ICP system, a standard containing the highest concentration level that would be reported for samples is 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.
- 12.6.3 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).
- 12.6.4 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.
- 12.6.5 The ICP instruments use an intelligent rinse program. The intelligent rinse lengthens the rinse time whenever a sample result for a known problem analyte is above a set concentration.

## 12.7 Demonstration of Capabilities

- 12.7.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually.
- 12.7.2 IDOCs and on-going proficiency demonstrations are conducted as follows: Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample is typically the LCS spike level. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

**12.7.3** If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

## **12.8 Training Requirements**

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. Further details concerning the training program are described in SOP DV-QA-0024.

## **13.0 Pollution Control**

**13.1** It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

**13.2** Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

## **14.0 Waste Management**

**14.1** All waste will be disposed of in accordance with federal, state, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Safety Manual, and DV-HS-001P, *Waste Management Plan*.

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

**14.2.1** Acid solutions from ICP drain - Waste Stream J

**14.2.2** Metals waste potentially contaminated with Cat 1 radioactive materials – Waste Stream RJ

**Note:** Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

## **15.0 References / Cross-References**

**15.1** Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Third Edition and all promulgated updates, EPA Office of Solid Waste, through January 2008.

**15.1.1** Method 6010C, Revision 3, Update IV, February 2007.

**15.1.2** Method 6010D, Revision 4, Update V, July 2014.

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

15.3 Department of Defense Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

**16.0 Method Modifications:**

Item	Method	Modification
1	EPA 6010C	This procedure uses mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.
2	EPA 6010C	The alternate run sequence presented in Section 10.5.3 is consistent with method requirements. Additional QC (i.e., ICSEA) analyses were added to accommodate the CLP protocol requirements.
3	EPA 6010C	Method 6010 states 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." Because of the lack of definition for "concentration range around the calibration blank," the laboratory has adopted the procedure in EPA CLP ILMO4.0 for determining IECs,
4	EPA 6010C	Section 9.9 of Method 6010C states: "If less than acceptable accuracy and precision data are generated, additional quality control tests are recommended prior to reporting concentration data for the elements in this method." The dilution test helps determine if a chemical or physical interference exists. Because the laboratory sometimes does not have prior knowledge if the MS/MSD will be within criteria, the analyst may select to perform a dilution test on one sample in each preparation batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. In this procedure, matrix interference is determined by evaluating data for the LCS, MS/MSD, and serial dilutions. The laboratory must request documented, clear guidance when an unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

**17.0 Attachments**

- Attachment 1 Metals Analyzed by ICP and Reporting Limits
- Attachment 2 Matrix Spike and Aqueous Laboratory Control Sample Levels
- Attachment 3 Low Level ICV and CCV Spiking Levels
- Attachment 4 Interference Check Sample Concentrations
- Attachment 5 TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels
- Attachment 6 6500 Initial Calibration & Continuing Calibration Verification Standards

- Attachment 7 Summary of Quality Control Requirements
- Attachment 8 MSA Guidance
- Attachment 9 Troubleshooting Guide
- Attachment 10 Contamination Controls
- Attachment 11 DoD QSM 5.0 QC Acceptance Criteria

## 18.0 **Revision History**

Revision 5, dated 31 July 2016

- Annual review
- Minor formatting and language corrections throughout
- Removed references to AFCEE and USACE
- Added Section 3.8 reagent water definition
- Added six-month expiration for intermediate standards in Section 7.2.2
- Added clarification to Section 7.3.2 regarding new standard verification
- Added new Section 7.18 explaining daily LR standard
- Removed cooling requirement for water samples in Section 8.0
- Added information to MS/MSD Section 9.5 to reflect current policy
- Changed Section 9.9 to Section 9.6, renumbered other sections accordingly
- Added information to Section 9.6 regarding the MSA requirement for TCLP extract samples
- Created subsections 9.6.1 - 9.6.4
- Added language to Section 10.4.9 to clarify daily linear range requirements
- Added Section 11.2 referencing the ferric iron work instruction
- Added requirement to review all manually transcribed data at second level review (Section 11.11.3)
- Archived pre-2011 revision histories

Revision 4, dated 31 December 2015

- Added requirements for Method 6010D to the SOP
- Minor grammar and formatting corrections throughout
- Added list of IEC test analytes to Section 12.5.4
- Added Section 10.5.6 regarding mid-run recalibration
- Added Section 12.2.5 defining 6010D IDL studies

Revision 3, dated 31 July 2015

- Annual review
- Updated Section 7.4 for how to make the 5% HNO<sub>3</sub>/5% HCl solution
- Updated Section 12.2 for MDLV spike level to 2-4x MDL
- Updated Section 12.8.2 to use LCSs instead of ICVs for the DOC
- Reformatting throughout
- Removed reference to silica holding time
- Added Maintenance and troubleshooting sections
- Replaced Section 11.10 to match current practice
- Removed Section 12.2
- Removed Sections 1.3.1 and 1.3.2
- Added new Section 1.6
- Removed reference to glass beads in Section 6.2
- Corrected Reagent and Standard formulae throughout to agree with current practice

Revision 2, dated 31 July 31 2014

- Annual review

- Updated Section 6.1.3 to specify purity of argon gas
- Added statement to section 9.1.2 to reference DoD QSM 5.0 criteria in Attachment 11
- Removed references to preparation of oil/oily samples throughout the document as the lab no longer supports this digestion method
- Added references for prep methods to section 15
- Added DOD QSM 5.0 QC acceptance criteria as Attachment 11

Revision 1, dated 15 July 2013

- Annual review
- Removed section 1.7
- Added section 3.8
- Corrected formatting
- Added section 11.12
- Removed Attachment 8, renumbered attachments and fixed references to attachments throughout the document

Revision 0.3, dated 13 July 2012

- Annual Review
- Clarified soil preservation for ICP only analysis, Section 8
- Updated section 9.1, 10.1, 10.2, and 12.1 to reflect current practice
- Updated sections 10.4.6 and 10.4.10 to control calibration blanks to ½ the RL

Revision 0.2, dated 30 June 2011

- Added reference to DV-IP-0017 "Microwave Digestion" throughout document
- Added section 6.3 "Computer Software and Hardware"
- Removed Uranium from the ICSA/ICSAB tables in sections 7.4, 7.5, and 7.6
- Updated sections 7.14 and 7.15 to reflect current practices
- Updated the Acceptance Criteria in sections 9.4, 9.6, and 9.10
- Referenced the TestAmerica Denver Quality Assurance Manual in section 10.4.1
- Updated section 11 to reference corporate SOP CA-Q-S-005, "Calibration Curves" and Arizona Calibration Training spreadsheet
- Added IEC calculation to section 11

*Earlier revision histories have been archived and are available upon request.*

**Attachment 1****Metals Analyzed by ICP and Reporting Limits**

<b>ELEMENT</b>	<b>Symbol</b>	<b>CAS #</b>	<b>6010 Analyte</b>	<b>Reporting Limit (µg/L) Water</b>	<b>Reporting Limit (mg/kg) Soil</b>
Aluminum	Al	7429-90-5	X	100	10
Antimony <sup>trace</sup>	Sb	7440-36-0	X	10	1
Arsenic <sup>trace</sup>	As	7440-38-2	X	15	1
Barium	Ba	7440-39-3	X	10	1
Beryllium	Be	7440-41-7	X	1	0.1
Bismuth	Bi	7440-69-9		100	10
Boron	B	7440-42-8	X	100	10
Cadmium <sup>trace</sup>	Cd	7440-43-9	X	5	0.5
Calcium	Ca	7440-70-2	X	200	20
Chromium	Cr	7440-47-3	X	10	1
Cobalt	Co	7440-48-4	X	10	1
Copper	Cu	7440-50-8	X	15	2
Iron	Fe	7439-89-6	X	100	10
Lead <sup>trace</sup>	Pb	7439-92-1	X	9	0.8
Lithium	Li	7439-93-2	X	10	5
Magnesium	Mg	7439-95-4	X	200	20
Manganese	Mn	7439-96-5	X	10	1
Molybdenum	Mo	7439-98-7	X	20	2
Nickel	Ni	7440-02-0	X	40	4
Phosphorus	P	7723-14-0	X	3,000	300
Potassium	K	7440-09-7	X	3,000	300
Selenium <sup>trace</sup>	Se	7782-49-2	X	15	1.3
Silicon	Si	7631-86-9		500	50
Silver <sup>trace</sup>	Ag	7440-22-4	X	10	1
Sodium	Na	7440-23-5	X	1	100
Strontium	Sr	7440-24-6	X	10	1
Sulfur	S	7704-34-9	X	200	2
Thallium <sup>trace</sup>	Tl	7440-28-0	X	15	1.2
Thorium	Th	7440-29-1		15	15
Tin	Sn	7440-31-5	X	100	10
Titanium	Ti	7440-32-6	X	10	1
Uranium	U	7440-61-1		60	20
Vanadium	V	7440-62-2	X	10	2
Zinc	Zn	7440-66-6	X	20	2
Zirconium	Zr	7440-67-7		15	1

**Attachment 2****Matrix Spike and Aqueous Laboratory Control Sample Levels**

<b>ELEMENT</b>	<b>LCS Level (µg/L)</b>	<b>Matrix Spike Level (µg/L)</b>
Aluminum	2,000	2,000
Antimony	500	500
Arsenic	2,000	2,000
Barium	2,000	2,000
Beryllium	50	50
Bismuth	2,000	2,000
Boron	1,000	1,000
Cadmium	50	50
Calcium	50,000	50,000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1,000	1,000
Lead	500	500
Lithium	1,000	1,000
Magnesium	50,000	50,000
Manganese	500	500
Molybdenum	1,000	1,000
Nickel	500	500
Phosphorous	10,000	10,000
Potassium	50,000	50,000
Selenium	2,000	2,000
Silicon	10,000	10,000
Si (as SiO <sub>2</sub> )	21,400	21,400
Silver	50	50
Sodium	50,000	50,000
Strontium	1,000	1,000
Sulfur	2,000	2,000
Thallium	2,000	2,000
Thorium	2,000	2,000
Tin	2,000	2,000
Titanium	1,000	1,000
Uranium	2,000	2,000
Vanadium	500	500
Zinc	500	500
Zirconium	500	500

**Attachment 3**  
**Low Level ICV/CCV**

<b>ELEMENT</b>	<b>LCS Level (µg/L)</b>
Aluminum	100
Antimony	10
Arsenic	15
Barium	10
Beryllium	1
Bismuth	100
Boron	100
Cadmium	5
Calcium	200
Chromium	10
Cobalt	10
Copper	15
Iron	100
Lead	9
Lithium	10
Magnesium	200
Manganese	10
Molybdenum	20
Nickel	40
Phosphorous	3,000
Potassium	3,000
Selenium	15
Silicon	500
Si (as SiO <sub>2</sub> )	1070
Silver	10
Sodium	1,000
Strontium	10
Thallium	15
Thorium	15
Tin	10
Titanium	10
Uranium	60
Vanadium	10
Zinc	20
Zirconium	15

**Attachment 4**

**Interference Check Sample Concentrations**

<b>Element</b>	<b>ICSA (µg/L)</b>	<b>ICSAB (µg/L)</b>
Aluminum	500,000	500,000
Antimony	-	1,000
Arsenic	-	2,000
Barium	-	500
Beryllium	-	500
Bismuth	-	1,000
Boron	-	2,000
Cadmium	-	1,000
Calcium	500,000	500,000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200,000	200,000
Lead	-	1,000
Lithium	-	1,000
Magnesium	500,000	500,000
Manganese	-	500
Molybdenum	-	1,000
Nickel	-	1,000
Phosphorous	-	2,000
Potassium	-	50,000
Selenium	-	5,000
Silicon	-	10,000
Silica	-	21,400
Silver	-	1,000
Sodium	-	50,000
Strontium	-	1,000
Sulfur	-	1,000
Thallium	-	10,000
Titanium	-	1,000
Vanadium	-	500

**Attachment 4**

**Interference Check Sample Concentrations (cont'd)**

<b>Element</b>	<b>ICSA (µg/L)</b>	<b>ICSAB (µg/L)</b>
Zinc	-	1,000
Tin	-	10,000
Thorium	-	10,000
Uranium	2,000	2,000
Zirconium	-	1,000

**Attachment 5**

**TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels**

<b>ELEMENT</b>	<b>Reporting Level (µg/L)</b>	<b>Regulatory Limit (µg/L)</b>	<b>Spike Level (µg/L)</b>
Arsenic	500	5000	4000
Barium	10000	100000	12000
Cadmium	100	1000	1100
Chromium	500	5000	5200
Lead	500	5000	5500
Selenium	250	1000	3000
Silver	500	5000	1050
Copper	100	N/A	2250
Zinc	200	N/A	2500

**Attachment 6**  
**6000 Dual View Calibration, ICV & CCV Standards**

<b>Element</b>	<b>Calibration Level</b>	<b>ICV (µg/L)</b>	<b>CCV (µg/L)</b>
Aluminum Lo	1,000	250	500
Aluminum Hi	100,000	40,000	50,000
Antimony	2,000	250	1,000
Arsenic	2,000	250	1,000
Barium	1,000	250	500
Beryllium	1,000	250	500
Bismuth	2,000	500	1000
Cadmium	1,000	250	500
Calcium	10,000	2,000	5,000
Chromium	1,000	250	500
Cobalt	1,000	250	500
Copper	1,000	250	500
Iron Lo	5,000	250	2,500
Iron Hi	100,000	80,000	50,000
Lead	2,000	250	1000
Magnesium	40,000	10,000	20,000
Manganese	1,000	250	500
Molybdenum	1,000	250	500
Nickel	1,000	250	500
Phosphorous	2,000	2,000	1,000
Potassium	100,000	20,000	50,000
Selenium	2,000	500	1,000
Silver	1,000	250	500
Sodium Lo	10,000	2000	5,000
Sodium Hi	500,000	40,000	250,000
Strontium	1,000	250	500
Sulfur	10,000	4,000	5,000
Thallium	2,000	500	1,000
Thorium	10,000	3,000	5,000
Tin	2,000	500	1,000
Vanadium	1,000	250	500
Uranium	20,000	5,000	10,000
Zinc	1,000	250	500
Zirconium	1,000	250	500

**Attachment 7  
 Summary Of Quality Control Requirements**

<b>QC Parameter</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between multiple exposures $\leq 5\%$	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
CCV	After the ICV, after every 10 samples and at the end of the run.	90-110% recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
RL Standard	At the beginning of the run	Results must within 50%	Terminate analysis; Correct the problem; Recalibrate.
LLICV/CCV	At the beginning of the run and after every 10 samples	Recovery must be within 30%	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable LLCCV.
ICB	Beginning of every analytical run, immediately following the initial CCV.	The result must be within $\pm 1/2$ RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCB	Immediately following each CCV (except for the CCV following the ICV).	The result must be within $\pm 1/2$ RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.10	See Section 9.10
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.10
Dilution Test	One per prep batch.	For samples $> 10x$ LOD (after dilution)' dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.

See Section 10.5.3 for run sequence to be followed.

**Attachment 7**

**Summary of Quality Control Requirements (Continued)**

<b>QC Parameter</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
Method Blank (MB)	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to ½ the RL.  Sample results greater than 10x the blank concentration are acceptable.  Samples for which the contaminant is < ½ RL may not require redigestion or reanalysis (see Section 9.3)	Re-run once in a clean tube. If > ½ RL, re-digest and reanalyze samples.  Note exceptions under criteria section.  See Section 9.4 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	LCS must be within 80 - 120% recovery or in-house control limits.  Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.4)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples.	75 – 125% recovery or tighter in-house control limits.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate (MSD)	One per sample preparation batch of up to 20 samples. 10% frequency for some programs (see 9.5)	75 – 125 % recovery; RPD ≤ 20% or tighter in-house control limits.	See Corrective Action for Matrix Spike.

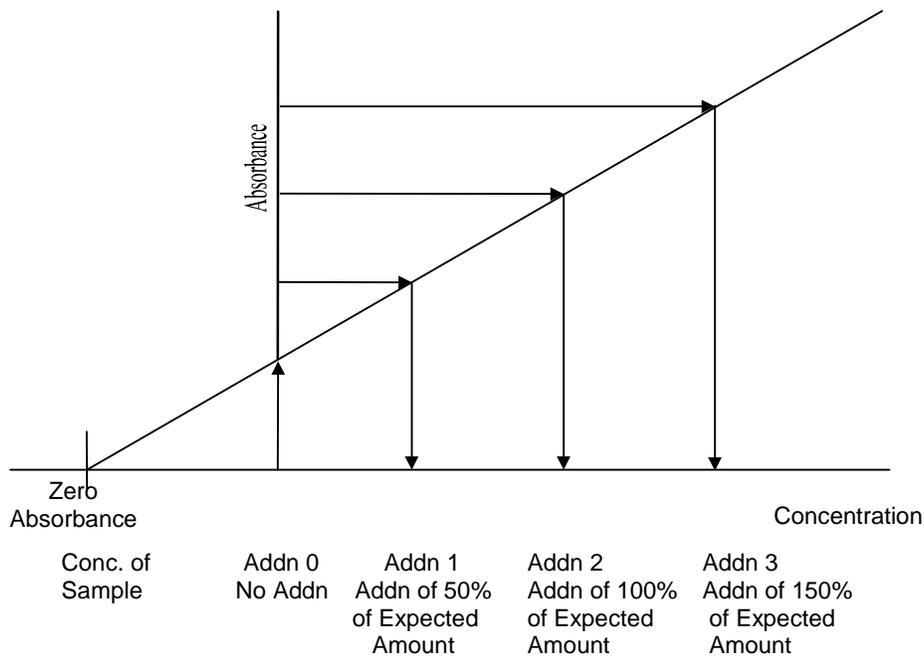
## Attachment 8

### MSA Guidance

#### Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the absolute value of 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 ( $r=0.995$  or greater) 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.

**Attachment 9**

**Troubleshooting Guide**

<b>Problem</b>	<b>Possible Cause/ Solution</b>
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer
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 Replace RF generator
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

## **Attachment 10**

### **Contamination Control Guidelines**

#### **The following procedures are strongly recommended to prevent contamination:**

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered gloves should not be used in the metals laboratory because the powder contains silica and zinc as well as other metallic analytes.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

#### **The following are helpful hints in the identification of the source of contaminants:**

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.

**Attachment 11**  
**DoD QSM 5.0 QC Criteria for Analysis by ICP**

<b>QSM 5.0 Table 8. Inorganic Analysis by ICP</b>	
<b>Requirement</b>	<b>DoD QSM 5.0 and DOE QSAS 3.0</b>
Linear Dynamic Range (LDR) or high-level standard check	<p>Run an LDR or high-level check standard at least once every 6 months. When calibrating with a single standard and a blank, the daily LDR standard must be analyzed at a concentration greater than any samples analyzed that day. Data cannot be reported above the high calibration range without an established/passing high-level check standard.</p> <p>Must be within <math>\pm 10\%</math> of expected value. Dilute samples within the calibration range or re-establish/verify the LDR.</p>
Initial Calibration (ICAL)	<p>Measure a minimum of one high standard and a calibration blank, daily. If more than one standard used, then <math>r^2 \geq 0.99</math> (<math>r \geq 0.995</math>), otherwise no acceptance criteria.</p> <p>The ICAL must pass before running any samples.</p> <p>NOTE: The laboratory currently performs duplicate burns for the ICPAES method.</p>
Initial Calibration Verification (ICV)	<p>Run second-source standard once after each ICAL and prior to sample analysis.</p> <p>All reported analytes must be within <math>\pm 10\%</math> of expected value.</p> <p>Correct any problems, verify standard, and rerun ICV. If that fails, correct problem and rerun ICAL. Verification must pass before running any samples.</p>
Continuing Calibration Verification (CCV)	<p>Run CCV after every 10 field samples, and at the end of the analysis sequence.</p> <p>All reported values within <math>\pm 10\%</math> of expected value</p> <p>If the CCV is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR).</p> <p>Correct any problems then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last successful CCV. Results cannot be reported without a valid CCV.</p> <p>Or</p> <p>Immediately (within one hour) analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.</p> <p>If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analytes(s) in all samples since the last acceptable CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p>
Low-Level Calibration Check Standard (Low-level ICV)	<p>Run low-level standard at a concentration <math>\leq</math> LOQ daily after one-point ICAL.</p> <p>All reported analytes must be within <math>\pm 20\%</math> of expected value.</p> <p>Correct any problems, then reanalyze or repeat ICAL. Results cannot be reported without a valid low-level calibration check standard.</p>

**Attachment 11**  
**DoD QSM 5.0 QC Criteria for Analysis by ICP**  
**(continued)**

<b>QSM 5.0 Table 8. Inorganic Analysis by ICP</b>	
<b>Requirement</b>	<b>DoD QSM 5.0 and DOE QSAS 3.0</b>
Initial and Continuing Calibration Blank (ICB.CCB)	<p>Analyze calibration blank before analyzing samples, after every 10 field samples, and at the end of the analysis sequence.</p> <p>No analytes detected &gt; ½ LOQ (RL) or &gt;1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. (13ICP) If not accepted by client, ICB/CCB must be &lt;LOD.</p> <p>If criteria not met, correct problem</p> <p>If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed. Correct any problems and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. CCB failures due to carryover may not require an ICAL.</p>
Interference Check Solution (ICS)	<p>Run the ICS at the beginning of an analytical run (after ICAL and prior to sample analysis).</p> <p>ICS-A: Absolute value of concentration for all non-spiked analytes must be &lt; LOD (unless they are a verified trace impurity from one of the spiked analytes).</p> <p>ICS-AB: Within ± 20% of expected value. (Note: ICS-AB not needed if instrument can read negative responses.)</p> <p>Correct any problems and reanalyze ICS. Do not analyze samples without a valid ICS.</p> <p>NOTE: TAL Denver has a letter from the ICSA standards manufacturer for many of the elements.</p>
Method Blank	<p>One per prep batch. No analytes detected &gt; ½ LOQ (RL) or &gt;1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common lab contaminants not detected &gt; LOQ. (2CLC)</p> <p>For ICP, common lab contaminants are: Al, Ca, Fe, K, Mg, Na, Si, Zn (Ba for TCLP)</p> <p>If criteria not met, correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.</p> <p>If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed.</p>
LCS	<p>One per prep batch. Recovery must meet DoD QSM limits.</p> <p>If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR).</p> <p>Correct any problems, then re-prepare and reanalyze LCS and associated samples for failed analytes in all samples in the associated batch. If corrective action fails, apply Q-flag to specific analyte(s) in all samples in associated batch.</p>

**Attachment 11**  
**DoD QSM 5.0 QC Criteria for Analysis by ICP**  
**(continued)**

<b>QSM 5.0 Table 8. Inorganic Analysis by ICP</b>	
<b>Requirement</b>	<b>DoD QSM 5.0 and DOE QSAS 3.0</b>
Matrix Spike (MS)	<p>One MS per prep batch. Use DoD acceptance criteria for LCS.</p> <p>If MS fails, consult project-specific DQOs and contact client to see if additional measures need to be taken.</p> <p>For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met.</p> <p>If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.</p>
MSD or Sample Duplicate	<p>Analyze one MSD or sample duplicate per prep batch per matrix. RPD between duplicates must be <math>\leq 20\%</math>.</p> <p>For failures, consult project-specific DQOs and contact client for additional measures to be taken.</p> <p>If acceptance criteria are not met, apply J-flag.</p> <p>If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.</p>
Dilution Test	<p>One per prep batch if MS or MSD fails. Only applicable for samples with concentrations <math>&gt;50x</math> LOQ (prior to dilution). For samples with lower concentrations perform PDS.</p> <p>Five-fold dilution must agree within <math>\pm 10\%</math> of the original result.</p> <p>Apply J-flag if acceptance criteria not met and explain in the case narrative.</p>
Post-Digestion Spike (PDS) Addition	<p>Perform Recovery Test when dilution test fails or analyte concentration in all samples is <math>&lt;50x</math> LOQ.</p> <p>Recovery must be within 80-120 % of expected result.</p> <p>If test fails, then run samples by MSA or apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 80 - 120%.</p>
Method of Standard Additions	<p>When dilution or post digestion spike fails <u>and</u> if required by the project. Document use of MSA in case narrative.</p>



***TestAmerica Denver***

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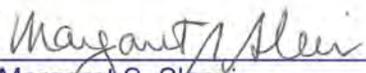
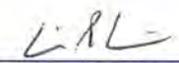
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Electronic Copy Only

## Title: Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series

### Approvals (Signature/Date):

 _____ Cheyana Cokley Technical Specialist	<u>6/26/17</u> Date	 _____ Adam Alban Health & Safety Manager / Coordinator	<u>28 June 17</u> Date
 _____ Margaret S. Sleevi Quality Assurance Manager	<u>6/28/17</u> Date	 _____ William S. Cicero Laboratory Director	<u>6/28/17</u> Date

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## 1.0 **Scope and Application**

- 1.1 This Standard Operating Procedure (SOP) is applicable to the solvent extraction of organic compounds from water samples, TCLP leachates, and SPLP leachates, using a separatory funnel. This SOP based on SW-846 Method 3510C, EPA 608, EPA 610, EPA 614, AK102, NWTPH-Dx, and Oklahoma DRO method.
- 1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.
- 1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, "Concentration of Organic Extracts", for details concerning the concentration and cleanup of extracts.

## 2.0 **Summary of Method**

A measured volume of sample, is placed in a separatory funnel. The pH is adjusted as required for the efficient extraction of specific compounds. The organic compounds are extracted with three portions of methylene chloride. The water phase is discarded. The organic phase is dried using sodium sulfate.

## 3.0 **Definitions**

Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

- 3.1 **Extraction Holding Time:** The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.
- 3.2 **Preparation Batch:** A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards
- 3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.
- 3.4 **Quality Assurance Summary (QAS):** Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

**3.5 Aliquot:** A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

**3.6 Reagent Water** (aka ELGA water – water generated from ELGA water polishing units): Water with a resistivity of 1 Megohm-cm or greater. The TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

#### **4.0 Interferences**

**4.1** Chemical and physical interferences may be encountered when analyzing samples using this method.

**4.2** Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

**4.3** Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented in an NCM.

**4.4** 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. Especially take note of the possibility of phthalate contamination from gloves. Gloves should be changed out frequently and whenever they come in contact with solvent. Glassware should be handled in a fashion that keeps gloves away from the interior and mouth of the glassware.

**4.5** The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenol may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510C. Method 3510C is preferred over Method 3520C for the analysis of these classes of compounds. However, the recovery of phenols is optimized by using Method 3520C and performing the initial extraction at the acid pH.

#### **5.0 Safety**

**5.1** Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

**5.2** 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, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

### 5.3 Specific Safety Concerns or Requirements

- 5.3.1** The use of separatory funnels to extract samples using methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity. Either a face shield must be worn over safety glasses or goggles must be worn when it is performed.
- 5.3.2** Glass centrifuge tubes can break in the centrifuge if proper care is not taken. This can lead to a hazardous material spill and endanger employees. Do not exceed the manufacturer's recommended maximum RPM for glass containers. Normally speeds greater than 2700 rpm are not advisable.
- 5.3.3** The procedure calls for the use of an electric rotator. The rotator is equipped with a safety latch that does not allow the rotator to rotate even if the power switch is turned on. The separatory funnels are secured to the rotator using straps. During the procedure it will be necessary to loosen the straps in order to un-stopper the separatory funnels. Whenever the straps are loose, the safety latch must be fastened to prevent the rotator from rotating.
- 5.3.4** Glasswool is a carcinogen and therefore should be handled in a hood to avoid inhalation of dust.

### 5.4 Primary Materials Used

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

#### Materials with Serious or Significant Hazard Rating

Material <sup>(1)</sup>	Hazards	Exposure Limit <sub>(2)</sub>	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.

Material <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and Symptoms of Exposure
Sodium Hydroxide	Corrosive Poison	2 mg/m <sup>3</sup>	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, and runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and can cause burns that may result in permanent impairment of vision, even blindness with greater exposures.
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.
Sulfuric Acid	Corrosive Carcinogen	1 mg/m <sup>3</sup>	Inhalation may cause irritation of the respiratory tract with burning pain the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.
<p>(1) Always add acid to water to prevent violent reactions.</p> <p>(2) Exposure limit refers to the OSHA regulatory exposure limit</p>			

## 6.0 Equipment and Supplies

**NOTE:** All glassware used in this procedure is cleaned following SOP DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use.

### 6.1 Supplies

- Separatory funnel, 2-liter with polytetrafluoroethylene (PTFE) stopcock and stopper.
- Separatory funnel, 500-mL with polytetrafluoroethylene (PTFE) stopcock and stopper.
- Separatory funnel rack and mechanical rotator.

- Balance,  $\geq 1400$  g capacity, accurate to  $\pm 1$  g, calibration checked daily per SOP DV-QA-0014.
- pH indicator paper, wide range.
- Class A Graduated Cylinder, sizes ranging from 50 mL to 1 L.
- Media bottles, 300 mL with Teflon-lined caps or capped with aluminum foil.
- Media bottles, 100 mL with Teflon-lined caps or capped with aluminum foil.
- Disposable pipettes, various volumes.
- Stemless glass funnel.
- Glass wool, baked at 400 °C for four hours.
- Mechanical pipette, 1 mL, positive displacement, with disposable tips, calibrated per SOP DV-QA-0008.
- Aluminum foil.
- Paper towels.

## **6.2 Computer Software and Hardware**

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

## **7.0 Reagents and Standards**

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

### **7.1 Reagent Water**

TestAmerica Denver has two ELGA water purification systems. The water coming from the ELGA system should be 18-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

## 7.2 Methylene Chloride

Each lot of solvent is tested following SOP CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

## 7.3 Acids and Bases

### 7.3.1 1:1 Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>), TALS Reagent ID "1:1 H<sub>2</sub>SO<sub>4</sub>"

Place an ice water bath on a stir plate. Place a container with a magnetic stir bar in the bath. While stirring, slowly add 1 part concentrated reagent grade sulfuric acid (36N) to 1 part water from the ELGA purification system. Assign a 1 year expiration date from the date made or the vender expiration date, whichever is shorter.

### 7.3.2 10N Sodium Hydroxide (NaOH), TALS Reagent ID "10N\_NaOH"

Purchased at ready-to-use concentration from commercial vendors. Assign a 1 year expiration date from the date opened or the vender expiration date, whichever is shorter.

### 7.3.3 1N Hydrochloric Acid (HCl), TALS Reagent ID "1N\_HCl"

Dilute 100 mL of stock reagent grade, concentrated HCl to 1000 mL with reagent water.

## 7.4 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400 °C oven for at least four hours. Store in tightly closed container.

## 7.5 Baked Sodium Chloride

Bake in 400 °C oven for at least 4 hours.

## 7.6 Standards

Please reference SOP DV-OP-00020 and WI-DV-009 for information regarding the surrogate and spike standards used in this procedure.

## 8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix and Method	Sample Container	Min. Sample Size	Preservation	Holding Time <sup>1</sup>	Reference
Water	Amber Glass	1000 mL	Cool, $\leq 6^{\circ}\text{C}$	7 Days	40 CFR Part 136.3
Water for Method AK 102	Amber Glass	1000 mL	Cool, $\leq 6^{\circ}\text{C}$ and $\text{pH} \leq 2$ with HCl	14 Days if properly preserved. 7 Days if un-preserved.	Method AK 102
Water for Method Oklahoma DRO	Amber Glass	1000 mL	Cool, $\leq 6^{\circ}\text{C}$ and $\text{pH} \leq 2$ with HCl	7 Days	Oklahoma Dept. of Environmental Quality
Water for Method NWTPH-DX	Amber Glass	1000 mL	Cool, $\leq 6^{\circ}\text{C}$ and $\text{pH} \leq 2$ with HCl	7 Days	NWTPH-Dx
Water for Method 8082 or 8082A	Amber Glass	1000 mL	Cool, $\leq 6^{\circ}\text{C}$	None <sup>2</sup>	SW-846 Chapter 4, Revision 4, Feb 2007
Water for Method 8081 or 8082 by Large Volume Injection	Amber Glass	250 mL	Cool, $\leq 6^{\circ}\text{C}$	7 Days	40 CFR Part 136.3
Water for Method 8270SIM by Large Volume Injection	Amber Glass	250 mL	Cool, $\leq 6^{\circ}\text{C}$	7 Days	40 CFR Part 136.3
TCLP Leachates	Glass	200 mL for 8270 100 mL for 8081 100mL for 8141	Cool, $\leq 6^{\circ}\text{C}$	7 Days from the start of the leach	SW-846 1311
SPLP Leachates	Glass	1000 mL	Cool, $\leq 6^{\circ}\text{C}$	7 Days from the start of the leach	SW-846 1312

<sup>1</sup> Exclusive of analysis.

<sup>2</sup> Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require a 1 week hold time for method 8082 and 8082A. The states of California, South Carolina, Pennsylvania, and Connecticut require a 1 week hold time.

## 9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Assurance Program*.

- 9.1.2** Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.
- 9.1.3** Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.
- 9.1.4** Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

## **9.2** Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

## **9.3** Method Blank (MB)

- 9.3.1** One method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.
- 9.3.2** The method blank for batches of aqueous samples for Large Volume Injection (prep method 3510C\_LVI) consists of 250mL of reagent water free of any of the analyte(s) of interest.
- 9.3.3** The method blank for batches of aqueous samples for all other methods consists of 1 L of reagent water free of any of the analyte(s) of interest.
- 9.3.4** The method blank for batches of TCLP leachates for methods 8081 and 8141 consists of 100 mL of leach fluid.
- 9.3.5** The method blank for batches of TCLP leachates for method 8270 consists of 200 mL of leach fluid.

**9.3.6** The method blank for batches of SPLP leachates consists of 1 L of leach fluid.

**9.4** Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

**9.4.1** At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

**9.4.2** The LCS for batches of aqueous samples for Large Volume Injection (prep method 3510C\_LVI) consists of 250mL of reagent water to which the analyte(s) of interest are added at known concentrations.

**9.4.3** For aqueous sample batches for all other methods, the LCS consists of 1 L of reagent water to which the analyte(s) of interest are added at known concentration.

**9.4.4** For methods 8081 and 8141 TCLP leachates, the LCS consists of 100 mL of leach fluid to which the analyte(s) of interest are added at known concentration.

**9.4.5** For method 8270 TCLP leachates, the LCS consists of 200 mL of leach fluid to which the analyte(s) of interest are added at known concentration.

**9.4.6** For SPLP leachates, the LCS consists of 1 L of leach fluid to which the analyte(s) of interest are added at known concentration.

**9.4.7** Method 608, 614, 610 requires a LCS at a 10% frequency. In other words one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

**9.4.8** Method AK102 requires LCS and a LCSD for every batch for every spike compound.

**9.5** Matrix Spike/Matrix Spike Duplicate (MS/MSD)

**9.5.1** One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

**9.5.2** If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared unless Method Comments indicate otherwise. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

**9.5.3** Method 608, 610, and 614 requires one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples. If there is insufficient sample volume for matrix spikes, then a LCSD must be performed.

**9.5.4** Method NWTPH-Dx requires a matrix spike and a matrix spike duplicate for every 10 samples. If insufficient sample volume is available for MS/MSD, a NCM must be written and a LCS and LCSD must be performed for every 10 samples.

## **9.6** Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

## **10.0** Procedure

**10.1** One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

**10.2** Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

**10.3** All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

## **10.4** Critical Procedural Considerations

**10.4.1** As stated throughout this SOP, analysts must review the Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).

**10.4.2** Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other separatory funnel than the designated one should be cleaned or disposed of before coming into contact with the sample.

**10.5** Assemble and clean the glassware immediately before use.

**NOTE:** Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

**10.5.1** Place a stopcock in each separatory funnel. For 1-liter extractions use a 2000 mL sepfunnel. For 250 mL, 200 mL and 100 mL extractions, use a 500 mL sepfunnel. Place a stopper for each separatory funnel on a clean sheet of aluminum foil that is marked with individual positions for each stopper. This is done to prevent cross-contamination.

**NOTE:** Samples logged with method 3510\_LVI are for Large Volume Injection methods and require 250 mL initial volumes. Samples logged for 8270 with a TCLP pre-prep require 200mL initial volumes. Samples logged for 8081 and 8141 with a TCLP pre-prep require 100 mL initial volumes.

**10.5.2** For each separatory funnel, plug a glass funnel with baked glass wool and add baked sodium sulfate. Place the funnel on a media bottle and place the media bottle below the separatory funnel.

**10.5.3** Rinse each separatory funnel once with methylene chloride. Be sure that all surfaces come into contact with the solvent. Drain the methylene chloride into the media bottle through the sodium sulfate.

**10.5.4** Rinse the sodium sulfate with additional methylene chloride if the first rinse did not completely saturate the sodium sulfate.

**10.5.5** Allow the methylene chloride to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional methylene chloride to the rinse if necessary.

**10.5.6** Discard the methylene chloride.

**10.5.7** Label each media bottle with the sample ID or batch QC ID.

## **10.6** Prepare LCS and Method Blank Samples

**NOTE:** For SW-846 methods if there is not a MS/MSD pair in the batch then perform a LCS/LCSD. Methods 608, 610, and 614 require a LCS and LCSD in batches of 11 samples or more or if there are no Matrix Spikes in batches of 10 or less.

**10.6.1** For aqueous sample batches logged for Large Volume Injection, (3510\_LVI), pour 250 mL of reagent water into the separatory funnels marked for the LCSs and the MB.

**10.6.2** For all other aqueous sample batches, pour 1 liter of reagent water into the separatory funnels marked for the LCSs and the MB.

**10.6.3** For 8270 TCLP leachates, use a 250 mL or 500 mL Class A graduated cylinder to measure out 200 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

- 10.6.4** For 8081 and 8141 TCLP leachates, use a 100 mL or 250 mL Class A graduated cylinder to measure out 100 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.
- 10.6.5** For SPLP leachates, use a 1000 mL Class A graduated cylinder to measure out 1000 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest 10 mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.
- 10.7** Measure the initial sample pH of the samples.
- 10.7.1** Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet.
- 10.7.2** If the sample is logged for AK102\_103, Okla\_DRO, or NWTPH\_Dx the samples should have been field preserved. See Section 8. If the samples are not preserved, an NCM should be written.
- 10.8** Aliquot the samples
- 10.8.1** For 8270 TCLP leachates, use a 250 mL or 500 mL Class A graduated cylinder to measure out 200 mL of the leachate. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.
- 10.8.2** For 8081 and 8141 TCLP leachates, use a 100 mL or 250 mL Class A graduated cylinder to measure out 100 mL of the leachate. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.
- 10.8.3** For SPLP leachates, use a 1 Liter Class A graduated cylinder to measure out 1000 mL of the leachate. Record the volume to the nearest 10 mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.
- 10.8.4** For water samples, it should be noted that TestAmerica Denver routinely aliquots gravimetrically. This is done to prevent cross-contamination due to volumetric glassware and to provide a more accurate initial volume measurement. However, some clients and regulatory programs require the laboratory to aliquot samples volumetrically. The Method Comments and QASs must be read before samples are aliquotted to check for this requirement. If samples are to be aliquotted volumetrically, use Class A volumetric glassware only and proceed to Section 10.8.6
- 10.8.5** Weigh the bottle (250 mL amber bottles for 3510C\_LVI or 1000 mL amber bottles for all other aqueous samples) and record the gross weight to the nearest gram. If there is any indication that the sample's density is not 1 g = 1 mL, then measure

the density of the sample using a calibrated pipette and an analytical balance. The weight of the sample extraction will be corrected for the density later. See Section 11 for the calculation. For example, normally a 1 liter bottle weighs 500 g when empty and when filled completely can only hold 1060 mL, therefore a full bottle weighing more than 1560 g is an indication that either the sample density is greater than 1g or the sample bottle contains a lot of sediment. Document any sample with a density greater than 1 g in an NCM.

**10.8.6** Inspect the samples for large amounts of sediment that may interfere with the extraction of the sample by causing excessive emulsions or clogging the stop-cock.

**10.8.6.1** If the sample contains so much sediment that the entire sample volume cannot be extracted, decant the sample into the separatory funnel (or a 1 L graduated cylinder if volumetric aliquotting is required), careful not to transfer the sediment. Write a NCM to document the sediment and that it prevented the entire sample volume from being extracted and the sample container from being solvent rinsed.

**10.8.6.2** If the sample does not contain a significant amount of sediment, then the entire sample volume will be used in the extraction. Do not pour the sample into the separatory funnel (or into the graduated cylinder if volumetric aliquotting is required) until after the surrogates and any necessary spikes have been added to the samples.

**10.8.6.3** For the 600 method series: if there is no more than an inch of sediment in the bottom of the sample bottle, shake the sample well and determine if the sediment resettles in approximately 1 minute. If not, the density of the sediment is likely to be low enough to stay suspended and not block the sidearm.

**10.8.6.4** For the 600 method series: if the density of the sediment is high and likely to cause a problem in the extraction or if there is more than an inch of sediment contact the PM so that the client's input can be obtained. Not extracting the entire sample and rinsing the bottle with the extraction solvent is a method deviation. If the client concurs that the sample can be decanted write an NCM to describe the deviation from the procedure.

**10.8.7** Place the sample containers in front of the separatory funnel labeled for that sample. A second analyst should then check the labels to make sure the correct sample is being extracted. This check is documented in the Organic Extraction Checklist (WI-DV-0009)

**10.9** Add Surrogates to All Field Samples and QC Samples

**10.9.1** The standards should be allowed to come to room temperature before spiking the samples. Record the ID of the standard used on the benchsheet.

**NOTE:** The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch Reference work instruction WI-DV-009 to determine the appropriate standard and the appropriate volume required.

**10.9.2** Only one batch should be surrogated at a time to ensure the correct standards are used.

**10.9.3** Add the appropriate volume of the appropriate working surrogate standard to the sample container for each sample and MS/MSD. Add the surrogate standard to the MB and the LCS's in the separatory funnels. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-009 to determine the appropriate standard and the appropriate volume required.

**NOTE:** If the sample contains an amount of sediment that has been deemed to interfere with the extraction process then the surrogate standard is added to the sample in the separatory funnel or in the graduated cylinder. This is considered a deviation and must be documented in a NCM.

**10.10** Add Spikes to all LCS's and MS/MSDs

**10.10.1** Add the appropriate volume of the appropriate working spike standard to the MS/MSD sample containers and the separatory funnels for the LCS and/or LCSD samples. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-009 to determine the appropriate standard and the appropriate volume required.

**10.11** Add approximately 6g (1 teaspoon) of NaCl to all samples and all QC samples. This is done to give the reagent water used in the MBs and LCSs some ionic strength to more closely mimic the matrix of actual water samples and to aide in the extraction of the more polar target compounds. Record the lot number of the sodium chloride on the bench sheet.

**10.12** If volumetric aliquotting is required, transfer the entire sample into a Class A graduated cylinder and record the volume on the benchsheet. If the sample bottle contains more than 1000 mL, a 100 mL Class A graduated cylinder can be used to complete the measurement. The entire sample volume must be used. Record the volume to the nearest 10 mL. Then pour the sample into the labeled separatory funnel. Place the used graduated cylinder in front of the appropriate separatory funnel so it can be solvent rinsed later.

**NOTE:** A 1000 mL Class A graduated cylinder is not accurate enough to measure to the nearest 1 mL. Therefore all samples that are aliquoted using a 1000 mL Class A graduated cylinder will have the initial volume recorded to the nearest 10 mL. This accuracy is sufficient.

**10.13** If volumetric aliquotting is not required, pour the sample directly into the separatory funnel. Place the empty sample container in front of the appropriate separatory funnel so it can be solvent rinsed.

**10.14** Adjust pH of Field Samples and QC Samples

Adjust the sample pH as indicated in the chart below using a minimum amount of 1:1 sulfuric acid (or 1 M hydrochloric acid for Methods AK102, Okla\_DRO and NWTPH\_Dx) or 10 N sodium hydroxide, as necessary. Record the adjusted pH and the lot number of the acid or base on the bench sheet. For TCLP leachates by method 8270, usually 1 mL of 1:1 sulfuric acid is sufficient.

**NOTE:** TCLP Leachates may have pH of < 5. In those cases, the pH should be adjusted per the table below.

Method	Initial Extraction pH	Secondary Extraction pH
All 8270 methods <u>except</u> SIM.	1 – 2	If samples are TCLP leachates extract at 14. If samples are water extract at 11 - 12
All 8270 SIM methods	As Received	None
All 8081, 8082 and 608 methods.	5 - 9	None
All 8141 and 614 methods	5-8	None
All 8015 methods	As Received	None
All 8310 and 610 methods	As Received	None
AK102_103 Okla_DRO NWTPH_Dx	If samples are preserved between pH 1 – 2, then acidify the MB and LCS. Otherwise extract as received and document insufficient preservation in an NCM.	None

**10.15** For 1 Liter samples, add 60 mL of methylene chloride to each empty sample container, unless the entire sample volume was not used. For 250 mL or smaller samples, add 30 mL of methylene chloride to each empty sample container, unless the entire sample volume was not used. Cap the container and shake gently to rinse all internal surfaces of the bottle. Pour the methylene chloride from the sample container into the appropriate separatory funnel. If a graduated cylinder was used to aliquot volumetrically, rinse the cylinder and add that rinse to the separatory funnel as well. Record the lot number of the methylene chloride on the bench sheet. If the sample contained significant sediment and the entire sample contents could not be extracted, do not rinse the empty sample container, but instead add the solvent directly to the separatory funnel. If the solvent rinse of the sample container cannot be performed, prepare a NCM.

**10.16** For water samples that were aliquotted gravimetrically, reweigh the bottle and calculate the initial sample volume by subtracting the empty bottles weight from the full bottles weight, assuming a density of 1 g = 1 mL. If there is any indication that the samples density is not 1 g = 1 mL then measure the density of the sample and correct the calculated initial volume accordingly using the formula in Section 11. Document abnormal sample density in an NCM. For example, normally a 1 liter bottle when filled completely

can only hold 1060 mL, therefore an initial volume greater than 1060mL is an indication that the density is not 1 g. Document any sample with a density greater than 1 g in an NCM.

**10.17** If the initial volume is less than 80% of the nominal volume, the sample reporting limits and method detection limits will be elevated substantially. Document this in a NCM.

**10.18** Stopper and rotate the separatory funnel for 3 minutes with periodic venting to release excess pressure. Document the extraction date and time on the benchsheet.

**WARNING:** Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken a few seconds. Vent into hood away from people and other samples. A face shield or goggles must be worn during venting.

**10.19** Allow the organic layer to separate from the water phase for at least 5 minutes or until complete visible separation has been achieved. This can take up to 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, use mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, pouring the solvent layer and emulsion back through the top of the separatory funnel (pour-back), or centrifugation. The emulsion could also be filtered through the glass funnel by adding additional sodium sulfate to remove all water in the emulsion. This technique should only be used after other techniques have failed to make complete phase separation and only after the last shake.

**NOTE 1:** If an emulsion forms, the analyst does not have to wait a complete 5 minutes before attempting to break the emulsion with pour-backs and centrifuge. Start employing the mechanical techniques right away to achieve phase separation.

**NOTE 2:** As much as 15 to 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

**10.20** Drain the lower methylene chloride layer into the sodium sulfate filled glass funnel. Allow the methylene chloride to drain completely into the media bottle. Rinse the sodium sulfate with a small amount of methylene chloride to ensure that all compounds of interest are collected in the media bottle. Record the lot number of the sodium sulfate on the bench sheet. If the sodium sulfate becomes saturated with water, add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

**10.21** Repeat the extraction two more times for a total of 3 extractions. Collect all three methylene chloride extracts in the same media bottle. For the 2<sup>nd</sup> and 3<sup>rd</sup> extractions it is not necessary to wait 5 minutes to allow the solvent to separate from the water; a 3 minute wait time should be sufficient.

**10.22** For the base/neutral and acid extractable method 8270, adjust the pH of the samples according to chart in Section 10.14. For 8270 TCLP leachates an excess of base is

required to effectively extract pyridine, therefore at least 7 mL of base should be used to ensure the pH is 14. Then extract the sample 3 more times. For these extractions, it is not necessary to wait 5 minutes to allow the solvent to separate from the water; a 3 minute wait time should be sufficient.

**NOTE:** For 8270 water extractions please note that typically 0.75mLs of acid is needed to achieve a pH 1-2; 2mL of base is typically required to achieve the pH of 11-12.

**10.23** Cap the media bottle with a Teflon-lined cap or aluminum foil and submit for concentration and possible clean-up steps.

**10.24** Dispose of the solvent-saturated water remaining in the separatory funnel in the appropriate waste container. See Section 14.

**10.25** Initial weights and volumes of samples are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-009).

**10.26** Troubleshooting

**10.26.1** If the sample appears very dark or viscous or in any way un-like water, stop and test the sample's miscibility before attempting to extract the sample by this procedure. Place a few milliliters of sample in a vial with methylene chloride. Cap and shake. If the sample is miscible in methylene chloride, the sample should be re-logged as a waste matrix with a prep method of 3580A.

**10.27** Maintenance

**10.27.1** Approximately every 6 months, the centrifuge should be lubricated.

**10.27.2** Contact the Facilities Manager immediately if the rotator is observed to be making un-familiar noises or rotating in a "jerking" manner.

## **11.0 Data Analysis and Calculations**

**11.1** Initial Volume calculation

$$InitialVolume(mL) = \frac{FullBottle(g) - EmptyBottle(g)}{Density(g / mL)}$$

**11.2** The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on DV-F-0045 Organic Extraction Department Checklist. See SOP DV-QA-0020 for more detail on the review process.

## **12.0 Method Performance**

### **12.1 Method Detection Limit Study (MDL)**

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

### **12.2 Limit of Quantitation Verification (LOQV)**

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

### **12.3 Demonstration of Capabilities**

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- 12.3.1** Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.
- 12.3.2** Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 12.3.3** If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- 12.3.4** Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.
- 12.3.5** Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

## 12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

## 13.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

## 14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

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

14.2.1 Methylene chloride – Waste Stream B

14.2.2 Solid waste/sodium sulfate – Waste Stream D

14.2.3 Basic aqueous sample waste saturated with methylene chloride – Waste Stream X.

14.2.4 Acidic aqueous sample waste saturated with methylene chloride – Waste Stream Y.

14.2.5 Neutral aqueous sample waste saturated with methylene chloride – Waste Stream X or Waste Stream Y.

14.2.6 Expired Standards/Reagents – Contact Waste Coordinator for guidance

**NOTE:** Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

## 15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005, Method

3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

- 15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, Organochlorine Pesticides and PCBs.
- 15.3 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 610, Polynuclear Aromatic Hydrocarbons.
- 15.4 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 614, Organophosphorous Pesticides.
- 15.5 Alaska Method AK102, “For the Determination of Diesel Range Organics”, Version 04/08/02.
- 15.6 Alaska Method AK103, “For the Determination of Residual Range Organics”, Version 04/08/02.
- 15.7 NWTPH-Dx “Semi-Volatile Petroleum Products Method for Soil and Water.
- 15.8 Oklahoma Department of Environmental Quality Methods 8000/8100 (Modified) Diesel Range Organics (DRO) Revision 4.1 Date 10/22/97
- 16.0 **Modifications:**
- 16.1 Modifications from SW-846 Method 3510C
  - 16.1.1 Section 7.1 of the method calls for initial sample volume to be determined volumetrically either by measuring out exactly 1 liter or marking the meniscus on the sample container and later determining the volume of water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware.
  - 16.1.2 Section 7.5 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.
  - 16.1.3 Section 7.6 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

- 16.1.4** The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.
- 16.1.5** The source method calls for samples to be extracted for method 8141 at the pH they are received. This procedure calls for the extraction to be performed at a pH between 5 and 8. This is done per guidelines found in Section 2 and Section 8 of SW-846 8141B.

## **16.2** Modifications from 40 CFR Method 608 and 610

- 16.2.1** Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.
- 16.2.2** Section 10.2 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.
- 16.2.3** Section 10.2 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.
- 16.2.4** Section 10.3 of the method calls for rinsing the sample collection bottle with the 60 mL methylene chloride aliquot for the second and third extraction as well as the first extraction. This SOP calls for rinsing the sample collection bottle with only the first 60-mL methylene chloride aliquot.
- 16.2.5** The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

## **16.3** Modifications from 40 CFR Method 614

- 16.3.1** Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.
- 16.3.2** Section 10.2 of the method calls for the extraction to be performed with at 15% v/v methylene chloride in hexane solvent. This procedure uses methylene chloride for the extraction. SOP DV-OP-0007 calls for the methylene chloride extract to be concentrated and exchanged to hexane.
- 16.3.3** Section 10.2 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.
- 16.3.4** Section 10.2 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic

layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

**16.3.5** Section 10.3 of the method calls for rinsing the sample collection bottle with the 60 mL solvent aliquot for the second and third extraction as well as the first extraction. This SOP calls for rinsing the sample collection bottle with only the first 60-mL methylene chloride aliquot.

**16.3.6** The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

#### **16.4** Modifications from Method AK 102

**16.4.1** Section 9.1.1.1 of the method calls for using no more than 1 liter of sample and to determine the volume either by measuring out exactly 1 liter or marking the meniscus on the sample container and later determining the volume of water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware. This SOP allows for the extraction of more than 1 L as it calls for the use of the entire sample volume.

**16.4.2** Section 9.1.1.6 of the method says to allow the water and solvent layers to separate for approximately 10 minutes. This SOP calls for the allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

**16.4.3** The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

#### **16.5** Modifications from Method NWTPH-Dx

**16.5.1** The method calls for determining the initial volume of the sample by marking the meniscus on the bottle and later determining the volume of tap water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware.

**16.5.2** The method calls for shaking the separatory funnel for one minute. This SOP calls for the separatory funnel to be shaken for at least three minutes.

**16.5.3** The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

## 16.6 Modifications from Oklahoma DRO

- 16.6.1** The method calls for aliquotting 800 mL to 900 mL of the sample volumetrically. This SOP calls for the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware. This SOP allows for the extraction of more than 1 L as it calls for the use of the entire sample volume.
- 16.6.2** The method calls for extracting using 50 mL of solvent. This SOP calls for the extraction to be done using at least 60 mL of solvent.
- 16.6.3** The method calls for shaking the separatory funnel for two minutes. This SOP calls for the separatory funnel to be shaken for at least three minutes.
- 16.6.4** The method calls for a method blank and LCS to be analyzed every 10 samples. This SOP calls for a method blank and LCS to be analyzed every batch of 20 samples.
- 16.6.5** The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

## 17.0 Attachments

Table 1. Determinative Methods Using Separatory Funnel Extractions

## 18.0 Revision History

- **Revision 15.0, June 30, 2017**
  - Paragraph with reference to QAM for basic definitions was added to Section 3.0
  - Removed previous Section 9.2 Initial Performance Studies, renumbered remaining Sections
  - Updated current Section 9.3.1 – removed “At least” for the one MB requirement
  - Added DoD requirements regarding MS/MSD (LCSD) to Section 9.5.2
  - Added current Section 10.2 regarding NCM documentation
  - The note was added to section 10.22 indicating the typical amounts of acid or base needed to achieve dual pHs for 8270 water extractions.
  - Added Section 11.2 regarding data review
  - Added current Section 12.2 LOQV information
  - Fixed numbering and section references throughout SOP
  - Removed Method 610 from Table 1. Lab no longer performing method.
- **Revision 14.0, June 30, 2016**
  - Added Section 3.6 – definition of reagent water
  - Revised the table in Section 8 to reflect the nominal leachate volume for method 8141.
  - Updated sections 9.4.4, 9.5.4, 10.4.1, and 10.5.4 to include 8141 TCLP.
  - Added Section 10.2 - recording of support equipment IDs

- Added note to Section 10.4 regarding the rotation of glassware
  - Added reference to method 8141 TCLP leachates to Section 10.7.2
  - Removed references to preparation of Wyoming Leachates throughout. Lab no longer performs Wyoming Leach method
  - Added Sections 10.7.6.3 and 10.7.6.4 to provide guidance for 600 method series in relation to sediment and decanting issues.
  - Updated Section 12 to be consistent with other SOPs
  - Renumbered paragraphs throughout due to the removal of the WY Leachate prep
- **Revision 13, August 31, 2015**
    - Annual Technical Review.
    - Removed the Notes from Section 2 and Section 10.9 regarding South Carolina. The laboratory no longer holds South Carolina certification for this method.
    - Added detail to Section 10.12 and 10.20 on how much acid and base is normally required to adjust the pH of leachates for method 8270.
- **Revision 12.0, August 31, 2014**
    - Revised Section 2 to remove references to initial volume. The procedure is used on waters and leachates with a variety of initial volumes. That detail is documented later in the procedure and was therefore removed from the summary found in Section 2.
    - Added a comment to Section 9.1.2 that states: "This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated."
    - Section 9 was revised to remove Acceptance Criteria and Corrective Action details. This information is found in the analytical procedures.
    - Removed the Note following Section 10.4.2 that instructs the analyst to check the samples for sodium thiosulfate preservation. TestAmerica Denver does not analyze drinking water samples by this procedure and therefore this preservation is not needed.
    - All references to 8270 by LVI were removed. TestAmerica Denver does not extract samples by this procedure for 8270 by LVI. Instead the samples are extracted by 3520C under DV-OP-0008.
    - The table in Section 10.12 was revised to make it easier to read and locate the correct Method.
    - Troubleshooting and Maintenance sections were added per DoD QSM 5.0 requirements.
- **Revision 11.0, August 19, 2013**
    - Added statement to Section 2.0 that LVI must not be used on SC samples
- **Revision 10.0, May 14, 2013**
    - The procedure was revised to instruct the analyst to allow the organic and aqueous phases to separate for a minimum of 5 minutes after the first extraction and 3 minutes after subsequent extractions.
    - The procedure was revised to increase the amount of sodium chloride added to samples and QC from 3g to 6g.
    - Section 5 was revised to include the hazards of glasswool and to instruct the analysts to handle it only in a fumehood.

- Section 8 was revised to change the hold-time calculation for leachates from the start of the leaching procedure instead of the completion of the leaching procedure. This was done to ensure the holding times are contiguous.
- Section 10.13 was revised to instruct the analyst to extract 250 mL to 100 mL samples with 30 mL of solvent instead of 15 mL of solvent. This was done to increase extraction efficiency while still reducing solvent usage.
- Sections 2.0, 9.1 and 10.1 were updated to reflect current practice.
- **Revision 9.0, January 15, 2013**
  - Section 10.9 was updated to include note to eliminate use of salt in South Carolina samples.
- **Revision 8.0, September 25, 2012**
  - This procedure was updated to include instructions on how to extract 8270 water samples for Large Volume Injection.
- **Revision 7.0, January 31, 2012**
  - Annual Technical Review
  - Updated Section 6.2 to describe the requirements for computer software and hardware
  - Updated Section 7.0 to describe requirements for Reagents and Standards.
  - Updated Section 8.0 to state PCBs by method 8082 have no holding time as per SW-846 Update 4 and that samples for analysis by NW-TPH have a 7 day hold time, even if acid preserved.
  - Updated Section 9.1.4 and Section 10.1 to accurately describe the NCM notification system.
  - Updated Section 10.4 and 10.6 to state the appropriate size of the graduated cylinders to be used to measure out 100 mL and 200 mL of leachate.
  - Updated Sections 10.6.6 and 10.14 to give guidance to the analyst when a density check of a sample is required.
  - Updated Section 10.9 to give more detail on how much sodium chloride should be added to the samples.
  - Updated Section 16 to include the method modification of the sodium chloride addition.
  - Updated Table 1 to reflect the current analytical SOPs.
  - Corrected grammatical and formatting errors
- **Revision 6.0 dated January 10, 2011**
  - Added note to Section 6 that sodium sulfate should be stored in tightly closed container.
  - Revised Section 7 to reference DV-OP-00020 for information about surrogate and spike standards.
  - Corrected Section 7.1 to indicate that the reagent water should be 18 to 18.2 Mohm/cm.
  - Revised procedure to include details on the extraction of Wyoming Leachates.
  - Added references to methods NWTPH-Dx, and Oklahoma DRO.

- Added Section 6.2 computer software and hardware.
- Section 8 was revised to give more detail on the preservation and hold times for methods AK102, AK103, NWTPH-Dx, and Oklahoma DRO.
- Revised Section 9 to include more detail on QC requirements for methods AK102\_103, NWTPH-Dx, and Oklahoma DRO.
- Revised Section 10 to clarify that when 1 liter graduated cylinders are used to measure the initial volume of the water samples, that the volume should be recorded to the nearest 10 mL.
- Revised Section 10 to instruct that if samples for methods AK102\_103, NWTPH-Dx, and Oklahoma DRO are received preserved, then the MB and the LCS samples should also be acidified with HCl. Otherwise the samples are extracted as received.
- Revised Section 16 to include more detail on modification from methods AK102\_103, NWTPH-Dx, and Oklahoma DRO
- Revised the procedure to call for the 2nd fraction of 8270 TCLP leachates to be extracted at a pH of 14 instead of the pH 11 to 12 used in water samples. This was done to help the recovery of pyridine.

*Earlier revision histories have been archived and are available upon request.*

**TABLE 1.**

**Determinative Methods Using Separatory Funnel Extractions**

<i>Method Description</i>	<i>Determinative Method</i>	<i>SOP</i>
Diesel Range Organics & Jet Fuels	SW-846 8015, California LUFT Method, Alaska Methods AK102 & AK103 SW-846 8015C	DV-GC-0027
Chlorinated Pesticides	SW-846 8081A SW-846 8081B EPA Method 608	DV-GC-0020 DV-GC-0016
Polychlorinated Biphenyls	SW-846 8082 SW-846 8082A EPA Method 608	DV-GC-0021 DV-GC-0016
Organophosphorus Pesticides	SW-846 8141A, & EPA Method 614	DV-GC-0017
Polynuclear Aromatic Hydrocarbons (PAH)	SW-846 8310	DV-LC-0009
Semi-volatiles by GC/MS	SW-846 8270 SW-846 8270D	DV-MS-0011 DV-MS-0012
PAH by GC/MS SIM	SW-846 8270	DV-MS-0002



**TestAmerica Denver**

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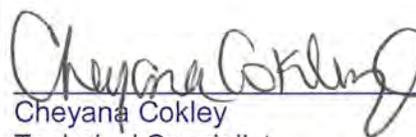
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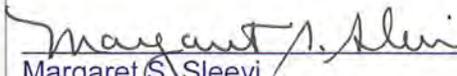
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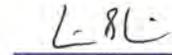
**Title: Concentration and Clean-up of Organic Extracts  
[SW-846, 3510C, 3520C,  
3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A, ASTM Method  
D7065-11, and EPA 600 Series Methods]**

**Approvals (Signature/Date):**

 10/19/16  
Cheyana Cokley Date  
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 19 Oct 16  
Adam Alban Date  
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## 1.0 **Scope and Application**

- 1.1 This standard operating procedure (SOP) provides instructions for the concentration, and if necessary, cleanup, of solvent extracts of organic compounds from water samples, soil samples, TCLP leachates, and SPLP leachates. This SOP is based on SW-846 Methods 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3630C, 3660B, 3665A, ASTM Method D7065-11, and EPA 600 Series methods.
- 1.2 The determinative methods and extraction methods used in conjunction with this procedure are listed in Attachment 1.
- 1.3 This procedure does not include the extraction steps. See the following SOPs for the applicable extraction procedures:

DV-OP-0006: Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C and EPA 600 Series

DV-OP-0008: Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C, and Method 625 and ASTM Method D7065-11

DV-OP-0010: Soxhlet Extraction of Solid Samples, SW-846 3540C

DV-OP-0015: Microwave Extraction of Solid Samples, SW-846 3546

DV-OP-0016: Ultrasonic Extraction of Solid Samples, SW-846 3550B and 3550C

DV-OP-0021: Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Low-Level NDMA by GC/CI/MS/MS

**NOTE:** This SOP does not include the concentration steps of extracts for Herbicides by method 8151A. See DV-OP-0011 instead.

## 2.0 **Summary of Method**

Sample extracts are concentrated to a specific final volume using an S-EVAP, N-EVAP, or Turbo-Vap. Some methods require a solvent exchange. If necessary, various clean-up techniques are performed before the extract is sent for analysis.

## 3.0 **Definitions**

Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

- 3.1 Extraction Holding Time:** The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.
- 3.2 Preparation Batch:** A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.
- 3.3 Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. See WI-DV-0032
- 3.4 Quality Assurance Summary (QAS):** Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/Method Comments field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

#### **4.0 Interferences**

Chemical and physical interferences may be encountered when analyzing samples using this method.

- 4.1** Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2** Visual interferences or anomalies (such as foaming, emulsions, odor, more than one layer of extract, etc.) must be documented.
- 4.3** 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.
- 4.4** Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by 8270D\_SIM\_LL must be concentrated in glassware designated for that method. K-D flasks, concentrator tubes, stem-less glass funnels, and Snyder columns will be clearly marked and segregated for this purpose.

#### **5.0 Safety**

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, 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, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

## 5.1 Specific Safety Concerns or Requirements

**5.1.1** In order to limit the emission of methylene chloride, TestAmerica Denver uses a solvent recovery system. The system condenses and collects methylene chloride that has been evaporated off the sample extracts while on the S-EVAP.

**5.1.1.1** Each analyst must inspect the system before using it to ensure the collection tubes are in good condition, the in-process tanks are not full, and the chiller is operating correctly.

**5.1.1.2** While concentrating methylene chloride or methylene chloride / acetone extracts on the S-Evap, the analyst must check the level of the solvent collected in the in-process tanks at a frequency to ensure the tank will not be overfilled. A tank will not be filled more than 90%. The analyst may use a timer set at 30 minute intervals to help remind the analyst to check the level of the solvent collected in the in-process tanks.

**5.1.1.3** The solvent recovery system will never be used for the collection of ether due to the potential danger to analysts if the system were to fail during operation.

**5.1.2** Glasswool is a carcinogen and therefore should be handled in a hood to avoid inhalation of dust.

## 5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit <sup>(1)</sup>	Signs and Symptoms of Exposure
Acetonitrile	Flammable Irritant Poison	40 ppm TWA	Exposure may cause cyanide poisoning resulting in reddening of the skin and eyes and pupil dilation. Effects of overexposure are often delayed due to the slow formation of cyanide ions in the body. May cause nose and throat irritation, flushing of the face, tightening of the chest. Also may cause headache, nausea, abdominal pain, convulsions, shock.
Hexane	Flammable	50 ppm TWA	Causes irritation to eyes, skin and respiratory tract. Aspiration hazard if swallowed. Can

Material	Hazards	Exposure Limit <sup>(1)</sup>	Signs and Symptoms of Exposure
	Irritant		enter lungs and cause damage. May cause nervous system effects. Breathing vapors may cause drowsiness and dizziness. Causes redness and pain to the skin and eyes.
Methanol	Flammable Irritant Poison	200 ppm TWA	Methanol evaporates at room temperature. Inhalation, ingestion and/or eye and skin contact can all possibly cause light-headedness, nausea, headache, and drowsiness. Prolonged exposure can lead to permanent blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Mercury	Corrosive Irritant Highly Toxic	0.05 mg/m <sup>3</sup> TWA	May be fatal if inhaled. May cause respiratory tract irritation.  May be harmful if absorbed through skin. May cause skin irritation.
Methylene Chloride	Irritant Carcinogen	25 ppm TWA 125 ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

## 6.0 Equipment and Supplies

**NOTE 1:** All glassware used in this procedure is cleaned following SOP# DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use. Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

**NOTE 2:** Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA method 8270D\_SIM\_LL and PAHs by method 8270C\_SIM\_LL must be concentrated in glassware designated for that method. K-D flasks, glass funnels, concentrator tubes, and snyder columns will be clearly marked and segregated for this purpose.

- 6.1 All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.
- 6.2 Kuderna-Danish (K-D) flasks.
- 6.3 Concentrator tubes for K-D flasks, un-graduated, approximately 10 mL.

- 6.4 Concentrator tubes for K-D flasks, graduated at 1mL, calibration checked before use following the steps detailed in DV-QA-0008.
- 6.5 Snyder columns, 3-ball with ground glass joints at top and bottom
- 6.6 Manual, adjustable positive-displacement pipette and bottle-top re-pipettor, used to dispense 1 to 20 mL. Calibration is checked following the steps detailed in DV-QA-0008.
- 6.7 Extract Storage Vials – variety of sizes, clear and amber
- 6.8 Pasteur pipettes – 6 inch and 9 inch in length.
- 6.9 Stem-less glass funnels
- 6.10 Glass wool, baked at 400°C for four hours.
- 6.11 Boiling Chips – contaminant free, approximately 10/40 mesh Teflon®, PTFE. For concentrating extracts to a final volume greater than 1mL.
- 6.12 Boiling Chips – contaminant free, carborundum #12 granules, for concentrating extracts to a 1mL final volume. These boiling chips are sufficiently small as to not add any error to the 1mL final volume.
- 6.13 Solvent Recovery System – includes re-circulating chiller, set at 5°C, cooling condensers, Teflon® PTFE tubing and In-Process Tanks with quick-connect attachments
- 6.14 S-Evap, thermostat controlled water bath
- 6.15 N-Evap, thermostat controlled water bath with regulated nitrogen supply
- 6.16 **Computer Software and Hardware**

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

## 7.0 **Reagents and Standards**

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

### 7.1 Methylene Chloride

Each lot of solvent is tested following CA-Q-S-001 or before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, that have not been previously tested per CA-Q-S-001, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

### 7.2 Hexane

For solvents packaged in bottles, each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas. For solvents packaged in CYCLETAINERS, the first batch of samples prepared with a new lot of solvent is monitored and reported to the QA group per the instructions in CA-Q-S-001 DV-1. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

### 7.3 Methanol, HPLC Grade

Each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

### 7.4 Acetone

Each lot of solvent is tested following CA-Q-S-001 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

### 7.5 Acetonitrile

Each lot of solvent is tested following CA-Q-S-001 DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

### 7.6 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400°C oven for at least four hours.

### 7.7 Sulfuric Acid, Concentrated

For use in PCB extract clean-up.

### 7.8 Florisil Solution, (FlorisilSol)

Add 900mL of hexane to a Class A graduated cylinder. Add 100 mL of Acetone to the same graduated cylinder for a final volume of 1000 mL. Pour the mixture into a 1 L amber bottle.

**7.9** Florisil Cartridges,

Purchased ready to use. 1000 mg in 6 mL tube. Stored in a desiccator after opening. Restek part number 24034 or equivalent.

**7.10** Anhydrous Silica Gel, 60-100 mesh, (SiGel60-100UA)

Sigma Aldrich part number 23799-1KG or equivalent

**7.11** Activated Anhydrous Silica Gel, 60-100 mesh, (Active SilGel)

Bake Silica Gel from Section 7.10 above at 400°C for at least 4 hours. Store in a desiccator.

**8.0** **Sample Collection, Preservation, Shipment and Storage**

**8.1** Sample extracts waiting to be concentrated are stored refrigerated at 0°C - 6°C in glass bottles or flasks and capped with Teflon-lined lids or aluminum foil. Final sample extracts are stored in glass vials with Teflon-lined lids. See Table 3 for details on storage vial types. Final concentrated extracts are stored refrigerated at 0°C - 6°C. Extracts have a holding time of 40 days from the date of extraction to the date of analysis.

**8.2** All sample extracts, before or after concentration, are stored separately from standards.

**9.0** **Quality Control**

**9.1** The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

**9.1.1** The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

**9.1.2** Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

**9.1.3** Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

**9.1.4** Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

## **9.2** Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12.0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

## **9.3** Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

## **9.4** Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of reagent water, and for batches of soil samples, consists of Ottawa sand, both of which are free of any of the analyte(s) of interest. The method blank for batches of TCLP and SPLP leachates consists of leach fluid. The method blank is processed and analyzed just as if it were a field sample.

## **9.5** Laboratory Control Sample (LCS)

**9.5.1** At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which the analyte(s) of interest are added at known concentration. For soil sample batches, the LCS consists of Ottawa sand to which the analyte(s) of interest are added at a known concentration. For TCLP and SPLP leachates, the LCS consists of leach fluid to which the analyte(s) of interest are added at known concentration. The LCS is carried through the entire analytical procedure just as if it were a sample.

**9.5.2** EPA Methods 608, 610, 614, and 625 require a LCS at a 10% frequency. In other words, one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

## 9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

- 9.6.1** One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.
- 9.6.2** EPA Methods 608, 610, 614, and 625 require one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples.
- 9.6.3** If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

## 9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

## 10.0 Procedure

- 10.1** One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.
- 10.2** Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

## 10.3 Critical Procedural Considerations

**NOTE:** Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

- 10.3.1** As stated throughout this SOP, analysts must review Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009).
- 10.3.2** Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces should be

cleaned or disposed of before coming into contact with the sample.

- 10.3.3** According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to WI-DV-0009 for the appropriate final solvents and final volumes.
- 10.4** Refer to WI-DV-0009 to determine if the extract is to be concentrated by the Kuderna-Danish / N-Evap method described in Section 10.5 and 10.6, or the Turbo-Vap method described in Section 10.6.6
- 10.5** Concentration by the Kuderna-Danish Method (S-evap)
- 10.5.1** Refer to WI-DV-0009. If the extract is to be concentrated to a 1 mL final volume, use a 1 mL graduated concentrator tube. For extracts that are to be concentrated to any other final volume, use an un-graduated concentrator tube.
- 10.5.2** Assemble the Kuderna-Danish concentrator by attaching the appropriate concentrator tube to the 500 mL K-D flask with a clip. Make sure the attachment is firm at the joint. While wearing cut-resistant gloves, tighten the joint with your fingertips and thumb. Do NOT over-tighten. Refer to Attachment 3 for configuration of the Kuderna-Danish concentrator.
- NOTE:** Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by method 8270D\_SIM\_LL and PAHs by method 8270C\_SIM\_LL must be concentrated in glassware designated for those methods. K-D flasks, concentrator tubes, and Snyder columns will be clearly marked and segregated for this purpose.
- 10.5.3** Rinse the apparatus with methylene chloride. Discard the rinse solvent into the appropriate waste container. Care should be taken to ensure all surfaces of the glass are coated with solvent.
- 10.5.4** If the extract is to be concentrated to a 1 mL final volume, add 2-3 carborundum granules to the K-D concentrator. If the extract is to be concentrated to a final volume greater than 1 mL, add 1-2 Teflon® boiling chips to each K-D concentrator.
- 10.5.5** If the sample extracts have not been filtered through sodium sulfate at the time of extraction, or if the sample extract have visible water, then the extracts must be dried at this point. Plug a glass funnel with baked glass wool and add approximately 1 teaspoon of baked sodium sulfate. Rinse the funnel and the sodium sulfate with methylene chloride and place it on top of the K-D. During the quantitative transfer in section 10.5.6 the extract will be filtered through the sodium sulfate.

**NOTE 1:** Glass wool dust is a carcinogen and therefore glass wool should only be handled in a hood to avoid inhaling any glass particles. Once covered with sodium sulfate, it can be removed from the hood.

**NOTE 2:** If the extract contains more water than can be easily removed by filtering through 1 teaspoon of sodium sulfate, either more sodium sulfate can be used or a solvent-rinsed separatory funnel can be used to separate the water out of the extract. A NCM should be prepared if this is necessary.

**10.5.6** Quantitatively transfer the sample extract to the K-D flask. Transfer the sample label to the K-D flask. Perform a quantitative transfer of the extract by rinsing the sample extract container with methylene chloride and adding the rinse solvent to the K-D. If the extract is being filtered through sodium sulfate, be sure to rinse the sodium sulfate well to ensure no target compounds are left on the sodium sulfate. Allow the solvent to drain from the sodium sulfate into the K-D flask then discard the sodium sulfate.

**10.5.7** Turn a three-ball Snyder column upside down and rinse with methylene chloride, then rinse the bottom joint with methylene chloride. Attach the Snyder column to the top of the K-D concentrator as shown in Attachment 3.

**10.5.8** Place the K-D concentrator on a s-evap water bath so that the tip of the receiver tube is submerged. The water level should not reach the joint between the concentrator tube and the K-D flask. Refer to WI-DV-0009 for the correct water bath temperature. Record both the observed and the corrected temperature on the benchsheet.

**10.5.9** For extracts that are methylene chloride or 50/50 methylene chloride/acetone, attach the solvent recovery system tube to the top of the Snyder column. At the appropriate rate of distillation, the balls will actively chatter but the chambers should not flood.

**NOTE 1:** For extracts for analysis for low-level NDMA by method 8270D\_SIM\_LL and PAHs by 8270C\_SIM\_LL, the solvent recovery system will not be used to avoid possible contamination.

**NOTE 2:** At this time, a timer may be set for no longer than 30 minutes as a reminder to check the in-process solvent tanks.

**10.5.10** If the method does not require a solvent exchange, skip to Section 10.5.12. If the method requires a solvent exchange, continue on to Section 10.5.11.

**10.5.11** If the method requires a solvent exchange at this time, detach the solvent recovery system tube from the top of the Snyder column and add the appropriate exchange solvent through the top of the Snyder column. The exchange solvent should be added when the extract has concentrated to a level that it forms a quarter-sized pool of solvent in the bottom of the K-D. Refer to WI-DV-0009 for details of exchange solvents and volumes. Mark the K-D flask and sample label to indicate the exchange has been performed. There is no need to re-attach the solvent recovery system at this time as the majority of the methylene chloride has already been evaporated and collected.

**10.5.12** Continue to concentrate the sample on the s-evap water bath back down to 10-15 mL, or just below the K-D and concentrator tube joint. At this point the boiling sample is just barely splashing above the top of the receiver tube.

**NOTE:** It is very important not to concentrate to dryness as analytes will be lost. Some of the analyses, especially for 8270 and 8015, are especially temperature sensitive and the sample should be taken off the water bath as soon as possible to avoid losing analytes. The 8081 surrogate TCMX is fairly volatile and can be lost if the extract is allowed to concentrate too low either before or after hexane exchange. If the analyst has concerns that the extract might have concentrated too low, they should notify their supervisor and/or write a NCM.

**10.5.13** Remove the K-D concentrator from the water bath. Rinse the Snyder column down with a minimal amount of solvent. If the extract was exchanged, use the exchange solvent to perform the rinse, otherwise use methylene chloride.

**10.5.14** Allow the extract to cool to room temperature, about 10 minutes.

**10.5.15** After the extract is allowed to cool, if the level of the extract is above the level of the concentrator tube joint, add a fresh boiling chip and return the K-D concentrator to the water bath.

**10.5.16** After the extract is cool, remove the snyder column. Remove the clip holding the K-D flask and concentrator tube together. Use a Kim-wipe to dry the water off of the joint area so that water does not get into the extract. Remove the concentrator tube from the K-D flask and rinse the lower K-D flask joint into the concentrator tube with methylene chloride or the appropriate exchange solvent.

## **10.6** Nitrogen Evaporation (N-Evap) to Final Concentration.

**10.6.1** N-evap needles should be cleaned weekly by soaking overnight in methylene chloride. This is documented in the N-evap needle log-book.

**10.6.2** At the beginning of each shift, the N-evap needles should be wiped clean with a Kim-wipe soaked in methylene chloride to remove any potential contamination. If a needle comes in contact with an extract, then it needs to be cleaned before being used on the next extract.

**10.6.3** Place the concentrator tube on the nitrogen evaporator. The temperature of the water bath should be at least 5°C below the boiling temperature of the solvent being evaporated (See Attachment 2). Lower the needle down to the sample so that a small dimple forms on the surface of the solvent. The stream of nitrogen should be gentle enough that it does not cause the extract to splash. Record both the observed and the actual temperature on the benchsheet.

**10.6.4** During the course of the evaporation, rinse the sides of the concentrator tube with approximately 1 mL of clean solvent. The rinse should occur when the solvent gets close to the final volume. Concentrate the solvent to just below the

final volume and remove from the nitrogen evaporator.

**10.6.5** Transfer the extract into the appropriate vial. Refer to WI-DV-0009 for the appropriate final volume and correct vial.

**10.6.5.1** If the extracts are to have a final volume of 1 mL, they should be in 1 mL graduated concentrator tubes. Using a Pasteur pipette, or a solvent wash bottle, add the appropriate solvent to the tube until the extract meniscus reaches the 1 mL gradation. Then using the Pasteur pipette transfer the extract to a labeled 2 mL amber glass vial.

**10.6.5.2** For extracts with a final volume greater than 1mL, the vials should be calibrated using the manual, adjustable positive-displacement pipette or bottle-top re-pipettor. Document the pipette ID used on the batch record. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract into the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.

**NOTE 1:** The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

**NOTE 2:** Some extracts might not concentrate down to the required final volume. If the extract is very dark and viscous, or an oil layer or precipitate starts to form, a higher final volume can be used. This should be documented in an NCM.

**10.6.6** After the extract has been transferred to the appropriate vial, rinse the concentrator tube with methylene chloride before washing per DV-OP-0004. This is important to remove any residual contamination.

## **10.7** TurboVap Method

**10.7.1** Turn on the TurboVap and adjust the water temperature to 40°C. Turn the nitrogen supply on. Record both the observed and the actual temperature on the benchsheet.

**10.7.2** Switch the endpoint sensor to "Manual".

**10.7.3** Adjust the water bath level. The water level should be at least 1 inch above the extract level.

**10.7.4** Turn on the nitrogen gas and adjust the gas pressure to approximately 12 psi. Lower pressure may be used if needed to prevent samples from splashing out of the TurboVap tubes.

- 10.7.5** Rinse the TurboVap tube with methylene chloride or the solvent the extract is in. Discard the waste.
- 10.7.6** Transfer the sample to the TurboVap tube. For 8141 soils extracted by soxhlet, dry the extract first by filtering through a funnel with baked sodium sulfate. Rinse the sample extract container with clean solvent and transfer to the TurboVap tube. Do not fill the TurboVap tubes over the fill line or approximately  $\frac{3}{4}$  full.
- 10.7.7** Place the TurboVap tube into the TurboVap and turn on nitrogen to the position the tube is in.
- 10.7.8** Close the lid. You should be able to see the sample extracts swirling in the tubes.
- NOTE:** If the extract splashes when the nitrogen flow starts, transfer a portion of the extract back into the original extract container, or lower the gas pressure.
- 10.7.9** As the extract concentrates, transfer the remainder of the extract in to the appropriate Turbovap tube. Rinse the sample container with a few milliliters of methylene chloride or appropriate solvent and transfer to the Turbovap tube.
- 10.7.10** During the concentration rinse the Turbovap tube walls with a few milliliters of solvent 1 or 2 times.
- 10.7.11** If a solvent exchange is required, concentrate to about 5 mL and add the exchange solvent. After the exchange solvent is added, swirl the extract to make sure the extract is well mixed. Concentrate back down to slightly less than the appropriate volume. Refer to Attachment 3 for details of exchange solvents and final volumes.
- 10.7.12** Transfer the extract into the appropriate vial.
- 10.7.12.1** Currently, the TurboVap is only used to concentrate extracts with final volumes greater than 1 mL. Ask the supervisor for guidance if a project requires a 1 mL final volume by TurboVap.
- 10.7.12.2** For extracts with a final volume greater than 1 mL, the vials should be calibrated using the manual, adjustable pipette or bottle-top re-pipettor. Document the pipette ID used on the batch record. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract to the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.
- 10.7.12.3** Rinse the Turbovap tube with methylene chloride 2-3 times before washing. Turbovap tubes are not baked. They are cleaned in

accordance with DV-OP-0004. If the Turbovap tubes need to be used again before they are dry, rinse with acetone to dry the Turbovap tube.

## 10.8 Cleanup Techniques

**NOTE:** If any sample in a batch requires a clean-up, the batch QC must also undergo the same clean-up technique.

### 10.8.1 Florisil Cartridge Cleanup

Florisil can be used to remove low-medium molecular weight polar hydrocarbon interfering compounds from pesticide extracts. The laboratory will use Florisil cleanups whenever water extracts have any color, whenever soil extracts have any color darker than a Post-It® Note, or whenever there is clear evidence of interferences, such as significant interfering peaks in the RT range for the target pesticide compounds or failing sample surrogate recoveries. Extracts that are to be analyzed for kepone will not be florisil cleaned, because florisil will remove kepone from the extract.

**NOTE:** Florisil cartridge performance checks are conducted for every lot of Florisil before use. Add 1.0 mL of the Florisil check solution described in Attachment 4 to a pre-rinsed Florisil cartridge. Following the procedure described below, load and elute the 1mL of check solution through the Florisil cartridge. Bring the final volume back down to 1.0 mL in hexane. The test sample must show 80-115 % recovery of the controlled analytes with < 5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected. The non-controlled analytes will be monitored for problems, but do not have to pass the 80-115% limits. If the check fails, repeat the test. If the re-check fails, contact QA for guidance.

#### 10.8.1.1 Clean the manifold and ports

Prior to each use, the top and underside of the manifold lid must be wiped down with hexane and a Kim-wipe to prevent any cross-contamination. The manifold ports must be left open and placed in a jar with fresh acetonitrile, in a sonication bath for a minimum of 30 minutes. The jar used in the soak and sonication of the ports must be replaced weekly to ensure it does not spread contamination. This is documented in the Organic Extraction Weekly Cleaning Logbook.

**10.8.1.2** Place one Florisil cartridge into the vacuum manifold for each sample extract. Make sure all valves are closed.

**10.8.1.3** Add approximately 6 mL of hexane to each cartridge by filling the tube.

**10.8.1.4** Slowly open the valves to allow a few drops of hexane to pass through, then close the valve and allow the hexane to soak the cartridge for at

least 5 minutes.

- 10.8.1.5** Slowly open the valves again and allow the hexane to drain through the cartridge but close the valve when the solvent level is right above the glass frit. Do not allow the cartridges to go dry. If cartridges go dry, repeat the conditioning step.
- 10.8.1.6** Remove the manifold top and place one clean, labeled 16 x 125 mm disposable glass test tube in each position for each of the samples. Replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.
- 10.8.1.7** Add exactly 2.0 mL of the concentrated extract to the appropriate Florisil cartridge. Turn the valve to the on position.
- 10.8.1.8** Allow the extract to gravity drip through the cartridge. The flow through the cartridges should be drop-wise, not streaming.
- 10.8.1.9** Just before the extract level drops below the glass frit, fill the cartridge with (90:10) Florisil solution. Allow this to pass through the cartridge, then just before it falls below the glass frit again, fill the cartridge again with (90:10) Florisil solution.
- 10.8.1.10** Allow all of the 90:10 solution to drip through the cartridges.

**NOTE:** Do not use the vacuum to recover solvent from the cartridge. If the vacuum is used and the cartridge goes dry under vacuum, then the interfering compounds that should be retained in the packing might come through into the cleaned extract.

- 10.8.1.11** Remove the tubes from the vacuum manifold and concentrate them back down to just below 2.0 mL on the nitrogen evaporator. Quantitatively transfer the extract to a 4mL vial that has been calibrated to hold 2.0 mL and bring the extracts up to the 2.0 mL calibration mark with hexane.
- 10.8.1.12** Discard the used cartridges.

## 10.8.2 Sulfur Removal

Sulfur can be removed by one of three methods: mercury, copper, or tetrabutylammonium sulfite (TBA), according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

#### 10.8.2.1 Sulfur Removal with Elemental Mercury

**NOTE:** Use Mercury in a hood and sparingly in order to minimize exposure and disposal costs.

**10.8.2.1.1** Transfer approximately 2 mL of sample extract into a clean Teflon-sealed vial.

**10.8.2.1.2** Add one to three drops of mercury to the extract vial and seal.

**10.8.2.1.3** Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.

**10.8.2.1.4** Remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

**10.8.2.1.5** If the mercury turns black, sulfur was present. Decant or pipette off the extract to a clean vial and repeat the procedure by adding one to three drops of fresh mercury. Do this until the mercury does not turn black.

**10.8.2.1.6** If the extract is cloudy, filter the extract through a 1µm disposable syringe filter.

**10.8.2.1.7** Properly dispose of the mercury waste.

#### 10.8.2.2 Sulfur Removal with Copper Powder

**NOTE:** This technique requires the copper powder to be very reactive, as demonstrated by a bright and shiny appearance. A pre-cleaned, activated copper may be purchased from a valid vendor. If manual preparation of reactive copper is performed, take care to remove all traces of acid in order to prevent degradation of some analytes.

**10.8.2.2.1** Weigh out copper into a 20 mL VOA VIAL assuming two grams of copper needed per sample.

**10.8.2.2.2** Remove oxides by treating with 10% nitric acid.

**10.8.2.2.3** Rinse the copper with DI organic-free water three times to remove all traces of acid.

**10.8.2.2.4** Rinse the copper with acetone and dry under a stream of nitrogen.

**10.8.2.2.5** Add approximately 2 grams of the copper powder to a 2 mL vial with approximately 1ml of sample extract and shake vigorously on a mechanical shaker for at least one minute.

**10.8.2.2.6** After phase separate, draw off extract and transfer to a clean vial.

### **10.8.3 Sulfuric Acid Cleanup**

**10.8.3.1** Add 1 mL of concentrated sulfuric acid to approximately 2 mL of sample extract in a Teflon capped vial.

**CAUTION:** There must be no water or acetone present in the extract or the reaction may shatter the sample container.

**10.8.3.2** Vortex for about 5 seconds and allow to settle. (Centrifuge if necessary)

**10.8.3.3** Remove the sample extract (top layer) from the acid using a Pasteur pipette and transfer to a clean vial.

**CAUTION:** It is not necessary to remove all the extract since the final volume is already determined. Transferring any amount of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column

**10.8.3.4** If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

**10.8.3.5** Properly dispose of the acid waste.

### **10.8.4 Silica Gel Clean-up for DRO extracts**

**10.8.4.1** Concentrate the DRO to slightly below 1 mL on the N-Evap. Add 100uL of the "SilicaGelSurr" standard to the extract and then bring the sample to a 1 mL final volume with methylene chloride.

**10.8.4.2** While the extract is still in the concentrator tube, add approximately 0.05 g of activated silica gel to the extract and mix with a Pasteur pipette.

**10.8.4.3** Transfer the extract to a new vial, leaving the silica gel behind. Then add a second aliquot of activated silica gel to the extract and mix by capping and shaking.

- 10.8.4.4** Allow the silica gel to settle out again and then transfer the extract to an empty vial and send on for analysis.

## 10.9 Documentation

All observations are recorded either directly into LIMS or on the hard-copy benchsheets. Any hand-written data recorded on the hard-copy benchsheets are transferred into LIMS before extracts are delivered to the analytical group. The hard-copy benchsheets are then saved and scanned into pdf files and sent to QA for archiving.

## 10.10 Maintenance

- 10.10.1** The chiller that operates the solvent recovery system should be checked periodically to ensure the water level is sufficient.
- 10.10.2** The SPE ports and valves used in the florisil are open and placed in a jar with fresh acetonitrile, in a sonication bath for a minimum of 30 minutes. The jar used in the soak and sonication of the ports must be replaced weekly to ensure it does not spread contamination. This is documented in the Organic Extraction Weekly Cleaning Logbook.
- 10.10.3** The N-Evap needles are removed once a week and soaked overnight in a jar of methylene chloride. This is documented in the Organic Extraction Weekly Cleaning Logbook.
- 10.10.4** The water bath used in the concentration of extracts has a thermostat that occasionally needs auto-tuned to keep the bath temperature within a narrow range. Record both the observed and the actual temperature on the benchsheet.

To start autotuning:

1. Press the **ⓂAdvance** key until the **[ AUE ]** prompt appears in the data display.
2. Select a thermal response value using the **ⓂUp-arrow/ⓂDown-arrow** keys: 1 for a slow response, 2 for an average response and 3 for a system that responds quickly. A thermal response value of 2 satisfactorily tunes most thermal systems.
3. Press the **ⓂAdvance** key. While the controller is in the tuning mode, the lower display alternately displays the normal information and the prompt **[ AUE ]**, at one-second intervals.

## 10.11 Troubleshooting

Unusual sample matrix may cause problems. If the extracts do not behave normally, contact a supervisor or senior analyst if you are unsure how to proceed. Document all observations and anomalies in a NCM.

## 11.0 Calibration

Not applicable to this procedure. See the determinative methods for calibration of the

analytical instrumentation.

## **12.0 Method Performance**

### **12.1 Method Detection Limit Study (MDL)**

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

### **12.2 Demonstration of Capabilities**

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

**12.2.1** Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

**12.2.2** Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

**12.2.3** If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

**12.2.4** Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

**12.2.5** Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

### **12.3 Training Requirements**

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

### **13.0 Pollution Control**

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

### **14.0 Waste Management**

**14.1** All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health & Safety Manual, and DV-HS-001P, "Waste Management Plan."

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

**14.2.1** Methylene chloride – Waste Stream B

**14.2.2** Flammable Solvents – Waste Stream C

**14.2.3** 1:1 MeCl<sub>2</sub>:Acetone – Waste Stream CA

**14.2.4** Solid waste/sodium sulfate – Waste Stream D

**14.3** Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

**NOTE:** Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

### **15.0 References / Cross-References**

**15.1** Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

**15.1.1** Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

**15.1.2** Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

**15.1.3** Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

- 15.1.4 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.
- 15.1.5 Method 3540C, Soxhlet Extraction, Revision 3, December 1996.
- 15.1.6 Method 3546, Microwave Extraction, Revision 0, February 2006.
- 15.1.7 Method 3620C, Florisil Cleanup, Revision 3, February 2007.
- 15.1.8 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.
- 15.1.9 Method 3660A, Sulfur Cleanup, Revision 1, July 1992.
- 15.1.10 Method 3665A, Sulfuric Acid/Permaganate Cleanup, Revision 1, December 1996.
- 15.1.11 Method 3630C, Silica Gel Cleanup, Revision 3, December 1996.
- 15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater
  - 15.2.1 Method 608, Organochlorine Pesticides and PCBs.
  - 15.2.2 Method 610, Polynuclear Aromatic Hydrocarbons.
  - 15.2.3 Method 614, The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater
  - 15.2.4 Method 625, Base/Neutrals and Acids.
- 15.3 ASTM D7065-11, Standard Test Method for Determination of Nonylphenols, Bisphenol A, p-tert-Octylphenol, Nonylphenol Monoethoxylate, and Nonylphenol Diethoxylate in Environmental Waters by Gas Chromatography Mass SpectrometryMethod Modifications:
- 16.0 **Modifications**
- 16.1 Method SW-846 3665A calls for the clean-up to be performed using 1:1 Sulfuric Acid:H<sub>2</sub>O. This procedure calls for the clean-up to be performed using concentrated sulfuric acid.
- 16.2 ASTM D7065-11 calls for the samples to be concentrated to a 0.5 mL final volume. This procedure calls for a 1 mL final volume.
- 16.3 Method SW-846 3620C calls for the florisil lot check to be performed using a standard containing the some pesticides at various concentrations from 5 ug/L to 50 ug/L. Per the source method, 1 mL of the standard is diluted to 2 mL (for concentrations between 2.5 ug/L and 25 ug/L) and the cleanup is then carried out and the cleaned extract concentrated to 1 mL for a final concentration of 5 ug/L to 50 ug/L. This procedure calls for the lot check to be performed using a standard containing all the pesticides at the same concentration of 50 ug/L. 1 mL of this standard is cleaned up without prior dilution

and then concentrated back down to 1 mL.

- 16.4** Method SW-846 3620C states that the florisil lot check passes if the pesticide recoveries are between 80% and 110% recovery. This procedure says the lot check passes if the pesticide recoveries are between 80% and 115%. This is done to match the CCV control limits.
- 16.5** Method SW-846 3620C states that the florisil lot check is to be performed using a standard containing the 2,4,5-Trichlorophenol at 0.1 ug/L. Per the source method, 0.5 mL of this standard is diluted to 2 mL (for a concentration of 0.025 ug/L) and the cleanup is then carried out and the cleaned extract concentrated to 1 mL for a concentration of 0.05 ug/L. This procedure calls for the lot check to be performed using a standard containing 2,4,5-trichlorophenol at 100 ug/L. 1 mL of this standard is cleaned up without prior dilution and then concentrated back down to 1 mL.
- 16.6** Method SW-846 3620C Section 11.1.3 states to condition the florisil cartridge with 4 mL of hexane. This procedure calls for 5 mL of hexane to be used. This is done for convenience.
- 16.7** Method SW-846 3630C calls for the silica gel clean-up to be performed with a column or SPE cartridge. This procedure calls for the silica gel to be added directly to the extract and mixed. The reverse surrogate used indicates if the clean-up is effective.

## **17.0 Attachments**

Attachment 1: Determinative and Extraction Methods Used in Conjunction with this SOP.

Attachment 2: Boiling Points of Solvents

Attachment 3: Kuderna-Danish Concentrator

Attachment 4: Florisil Check Solution

## **18.0 Revision History**

- Revision 11, dated 31 October 2016
  - Added the paragraph referencing the QAM for general definitions in Section 3.0
  - Added the requirement to document the ID of pipettes used in Sections 6.1, 10.6.5.2 and 10.7.12.2.
  - Updated Section 10.1 to reflect current practices
  - Added the specification of using the S-evap for concentration in Sections 10.5, 10.5.8 and 10.5.12
  - Added the requirement to document both the observed and actual temperature in Sections 10.6.3 and 10.10.4
- Revision 10 dated 31 December 2015
  - Updated formatting and numbering throughout the document
  - Revised method code references to reflect current practice
  - Numbered NOTES where there were multiples (Sections 6.0, 10.4.5, 10.4.9, 10.5.5.2)
  - Updated drive reference in Section 6.1
  - Updated "Reagent Grade Chemicals" definition in Section 7.0 to be consistent with other SOPs

- Added statement in Section 8 to specify that extracts are stored separately from standards
- Updated Section 9.1 to be consistent with other SOPs
- Added new section 10.2 for consistency with other SOPs
- Added NOTE to Section 10.3
- Added a requirement to Section 10.6.6 to rinse all concentrator tubes with methylene chloride before washing
- Removed the reference to South Carolina in Section 10.8.2. The laboratory no longer holds certification for South Carolina by this method
- Updated Section 12 to be consistent with other SOPs
- Added NOTE to Section 14.3
- Revised the concentration of 2,4,5-Trichlorophenol in the Florisil Check Solution described in Attachment 4.
  - The compound used to be at a concentration of 0.1 ug/mL in the standard
  - It is now at a concentration of 0.5 ug/mL
  - One mL of the standard is used in the Florisil check procedure, resulting in 0.5 µg of the compound loaded onto the 6 g of Florisil
- Removed references to DV-MS-0005 in Section 1 and Attachment 1, the laboratory no longer performs this procedure
- Revision 9 dated 31 December 2014
  - Section 5.1.1.2 and Section 10.4.9 were revised to match current practice on the use of the solvent recovery system.
  - Section 6.1 Computer Software and Hardware was added.
  - Section 7.6 Baked Sodium Sulfate was revised to match current practice and the latest revision of CA-Q-S-001 DV-1.
  - Section 7.11 was revised to correct the TAL Reagent ID.
  - Section 9.1 was revised to include the statement "This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated".
  - Section 9.4, 9.5, 9.6, and 9.7 were revised to remove information on Acceptance Criteria and Corrective Action. This information can be found in the analytical and QA SOPs.
  - Section 10.4.5 was revised to instruct the analyst to use approximately 1 teaspoon of sodium sulfate to dry extracts. This was done to limit the extract's exposure to sodium sulfate which can cause low recoveries for some acid compounds. A note was also added to this section to instruct the analyst to use more sodium sulfate or a separatory funnel to remove water if a teaspoon of sodium sulfate is not sufficient.
  - The Note in Section 10.4.12 was revised to instruct the analyst to write an NCM and/or notify their supervisor if they have a concern that an extract concentrated too low.
  - Section 10.7.1 Florisil Clean-up was revised to give guidance on what to do if the florisil check fails.
  - Section 10.7.1 was revised to instruct the analyst to not use the vacuum to pull all of the solvent from the cartridge. This was done to prevent interfering compounds and 2,4,5-TCP from eluting off of the cartridge.
  - Section 10.7.1 and Attachment 4 Florisil Check Solution were revised to indicate which compounds are controlled and which compounds are monitored. In addition, surrogate compounds were added to the solution.
  - Section 10.7.1 and 10.9 were revised to instruct the analyst to soak the SPE ports in a jar with the valves open instead of disassembling the valves.
  - Section 10.7.3 was revised to instruct the analyst to perform the clean-up on approximately 2mL of extract. This was done to match current practice.

- Section 10.7.4 Silica Gel Clean-up and Sections 15.1 and 16.0 were revised to match current practice.
- Section 10.9 Maintenance was revised to include instructions on how to tune the water bath thermostat.
- Attachment 3 – Concentration Summary was removed and replaced with WI-DV-0009. All other Attachments were re-numbered.
- Revision 8 dated 13 December 2013
  - The procedure was revised to include ASTM D7065-11.
  - The procedure was revised to include steps for silica gel clean-up for DRO extracts.
  - Section 7 was revised to include details on the Florisil Solution and Florisil cartridges. These details were lacking in previous revisions.
  - Section 10.4.2 was revised to give more detail on how to safely tighten the ground glass joint between the KD and concentrator tube.
  - Section 10.6.3 was revised to give more detail about the required water level in the Turbo-Vap.
  - Maintenance and Troubleshooting sections were added as Sections 10.8 and 10.9.
  - Section 16 was revised to include method modifications from SW-846 3620C.
  - Attachment 1 was updated to reflect the current SOPs in use in the laboratory.
  - Attachment 3 was updated.
- Revision 7 dated 5 December 2012
  - Section 5 and Section 10.4.5 were revised to instruct the analysts to handle glass wool in a hood to avoid breathing in the dust.
  - Revised Section 10.4.8 to instruct the analysts to document both the observed and corrected temperatures.
  - Section 10.7.1.11 was revised to describe in more detail how the florisiled extracts are taken to the 2 mL final volume.
  - Section 14.2 was revised to include the waste stream for 1:1 MeCl<sub>2</sub>:Acetone – Waste Stream CA.
  - Attachment 1 was revised to include DV-OP-0015 as an acceptable extraction for Diesel Range Organics.
  - Attachment 3 was revised to include details on 8081/3510\_LL concentration steps.
- Revision 6.0 dated 14 October 2011
  - The procedure was revised to remove instructions on how to concentrate and clean up extract for method 8070 and 607. TestAmerica Denver no longer supports these methods.
  - Section 1.3 was corrected to give the correct SOP number to Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Low-Level NDMA by GC/CI/MS/MS.
  - Section 7.5 was revised to state acetonitrile is tested before use. Previously this solvent was not tested before use.
  - The procedure was revised to include instructions that all extracts for analysis by method 8081, 8082, or 608 to be hexane exchanged only after concentration on the S-Evap. Previously the SOP instructed analysts to add the hexane exchange before the S-Evap for extracts that were concentrated by microwave extraction. This resulted in poor hexane exchanges, therefore the extracts are now concentrated before the exchange.
  - The procedure was revised to instruct analysts not to use the solvent recovery system when concentrating samples for analysis of low-level NDMA by

GC/CI/MS/MS. This was done to eliminate a possible source of contamination in this ppt level analysis.

- The procedure was revised to instruct analysts to use concentrated sulfuric acid in the acid clean up of PCB extracts.
- The procedure was revised to clarify the exact steps used in the sulfur removal with mercury.

*Earlier revision histories have been archived and are available upon request.*

**Attachment 1.**

**Determinative and Extraction Methods Used in Conjunction with this SOP**

<b>Method Description</b>	<b>Determinative Method</b>	<b>Determinative Method SOP</b>	<b>Extraction Method</b>	<b>Extraction Method SOP</b>
Diesel Range Organics & Jet Fuels	SW-846 8015B, 8015C, 8015D, California LUFT Method, & AK102 & AK103, NW-TPH, OK DRO	DV-GC-0027	WATER: SW-846 3510C, AK102 AK103 NW-TPH OK DRO SOIL: SW-846 3550B/C SW-846 3546 AK102, AK103 NW-TPH OK DRO	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Chlorinated Pesticides	SW-846 8081A, 8081B & EPA Method 608	DV-GC-0020 DV-GC-0016	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Polychlorinated Biphenyls	SW-846 8082, 8082A EPA Method 608	DV-GC-0021 DV-GC-0016	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Organo-phosphorus Pesticides	SW-846 8141A, 8141B, & EPA Method 614	DV-GC-0017	WATER: SW-846 3510C SOIL: SW-846 3540C	WATER: DV-OP-0006 SOIL: DV-OP-0010
Polynuclear Aromatic Hydrocarbons	SW-846 8310 & EPA Method 610	DV-LC-0009	WATER: SW-846 3510C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 SOIL: DV-OP-0016
Semi-volatiles by GC/MS	SW-846 8270C, 8270D & EPA 625	DV-MS-0011 DV-MS-0012	WATER: SW-846 3510C SW-846 3520C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 or DV-OP-0008 SOIL: DV-OP-0016
Low-Level Semi-Volatiles by GC/MS	SW-846 8270C	DV-MS-0011	WATER: SW-846 3520C	WATER: DV-OP-0008
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM	DV-MS-0002	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0008 SOIL: DV-OP-0016 or DV-OP-0015
Isotope Dilution Analysis of n-Nitrosodimethylamine by GCMS SIM using LVI	SOP	DV-MS-0015	WATER: SW-846 3520C SOIL: SW-846 3550B/C	WATER: DV-OP-0021 SOIL: DV-OP-0016

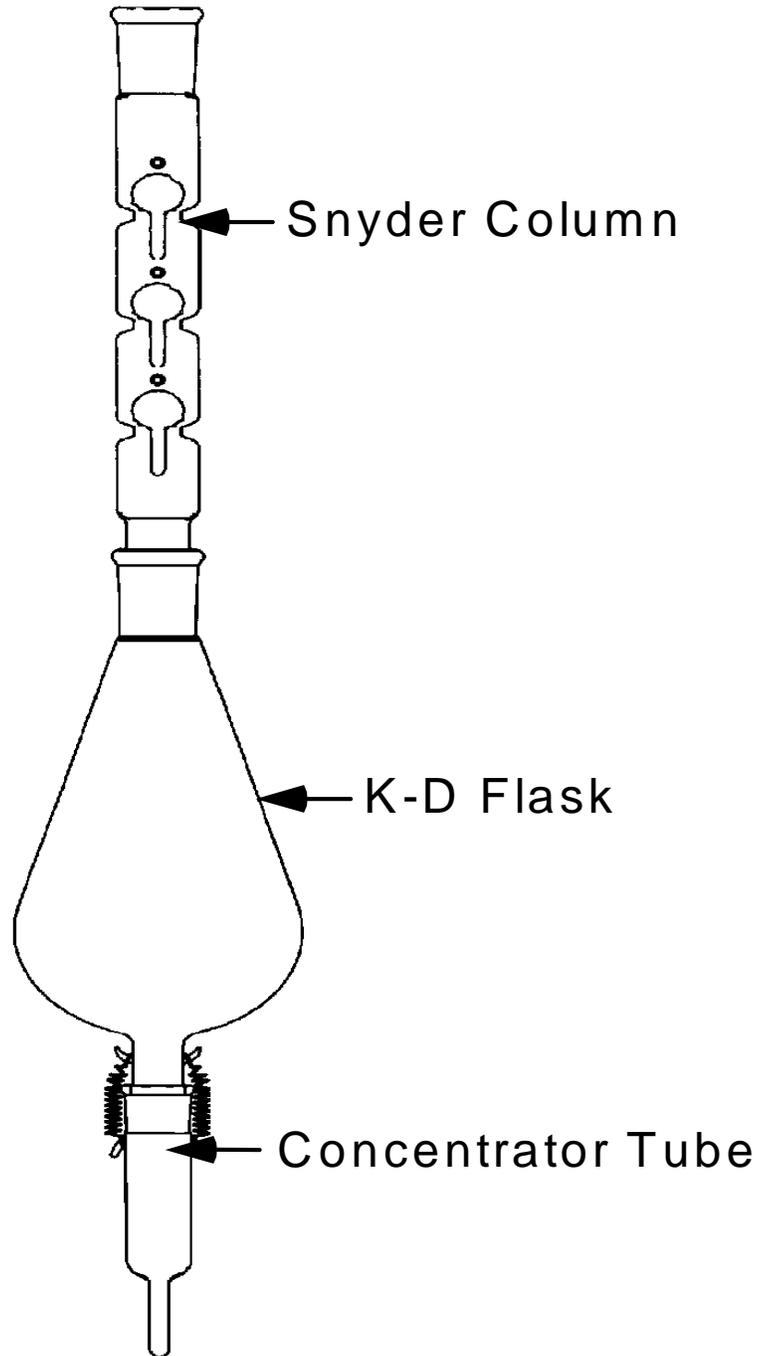
**Attachment 2.**

**Boiling Points of Solvents**

<b>Solvent</b>	<b>Boiling Point (°C)</b>
<b>Methylene chloride</b>	<b>40</b>
<b>Acetone</b>	<b>56</b>
<b>Hexane</b>	<b>69</b>
<b>Methanol</b>	<b>65</b>
<b>Acetonitrile</b>	<b>82</b>

**Attachment 3.**

**Kuderna-Danish Concentrator**



**Attachment 4.****Florisol Check Solution  
Prepared in Hexane**

<b>Compound</b>	<b>Concentration</b>	<b>Control</b>
2,4,5-Trichlorophenol	0.05ug/mL	Y
Alpha-BHC	0.05ug/mL	Y
Alpha-Chlordane	0.05ug/mL	N
Aldrin	0.05ug/mL	N
Beta-BHC	0.05ug/mL	N
Dieldrin	0.05ug/mL	Y
Endosulfan I	0.05ug/mL	Y
Endosulfan II	0.05ug/mL	N
Endosulfan sulfate	0.05ug/mL	N
Endrin	0.05ug/mL	Y
Endrin Aldehyde	0.05ug/mL	N
Endrin Ketone	0.05ug/mL	N
Gamma-BHC	0.05ug/mL	Y
Gamma-Chlordane	0.05ug/mL	N
Heptachlor	0.05ug/mL	Y
Heptachlor expoxide	0.05ug/mL	N
Methoxychlor	0.05ug/mL	Y
4,4-DDD	0.05ug/mL	Y
4,4-DDE	0.05ug/mL	N
4,4-DDT	0.05ug/mL	Y
Tetrachloro-m-xylene	0.02ug/mL	Y
Decachlorobiphenyl	0.02ug/mL	Y



**TestAmerica Denver**

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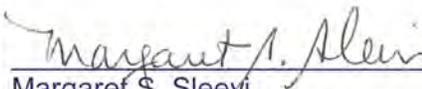
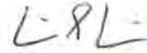
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Electronic Copy Only

## Title: Microwave Extraction of Solid Samples by Method [SW-846 3546]

Approvals (Signature/Date):	
 Cheyana Cokley Technical Specialist	<u>1/31/17</u> Date
 Adam Alban Health & Safety Manager / Coordinator	<u>31 Jan 17</u> Date
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## 1.0 Scope and Application

- 1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples using microwave energy to produce elevated temperature and pressure conditions in a closed vessel containing the sample and organic solvent. This procedure achieves analyte recoveries equivalent to those from soxhlet or sonications methods, but uses less solvent. This SOP is based on SW-846 Method 3546.
- 1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate solvents and spiking mixtures are used.
- 1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, Concentration of Organic Extracts, for those details.

## 2.0 Summary of Method

A measured weight of sample, typically 30 g, is solvent extracted using a microwave extractor.

## 3.0 Definitions

Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

- 3.1 **Extraction Holding Time:** The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.
- 3.2 **Preparation Batch:** A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards
- 3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 for details on Method Comments.
- 3.4 **Quality Assurance Summary (QAS):** Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

**3.5 Aliquot:** A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

#### **4.0 Interferences**

**4.1** Chemical and physical interferences may be encountered when analyzing samples using this method.

**4.2** Sodium sulfate is not used in the extraction vessel. This is because salts are known to super heat when exposed to microwave energy. Samples are extracted without the addition of sodium sulfate, but the extracts are dried with sodium sulfate after the extraction, before concentration of the extracts. If the sample is excessively wet the aliquot can be divided among two or three extraction vessels and the extracts combined prior to concentration.

**4.3** Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of this SOP (Section 9). Specific selection of reagents may be required to avoid introduction of contaminants.

**4.4** Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

**4.5** 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.

**4.6** Paint chips are an especially difficult matrix to extract. Oftentimes the paint chips dissolve or partially dissolve in solvents and therefore can ruin glassware and extraction vessels. It is the laboratory’s experience that paint chips are best extracted by method SW-846 3580 instead of 3550C or 3546.

#### **5.0 Safety**

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, 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.

## 5.1 Specific Safety Concerns or Requirements

- 5.1.1** A post-run cool down must be used after each extraction to prevent the possibility of operator burns. Pressure builds up in the closed vessel at high temperatures. Care should be taken when opening the vessel when it is above room temperature.
- 5.1.2** Samples that contain metal fragments or metal components of any kind should not be extracted by this procedure. These samples should be extracted by method SW-846 3550C instead. Care should be taken to inspect samples carefully as they are aliquotted.
- 5.1.3** Eye protection that satisfies ANSI Z87.1 (as described in the Corporate Safety Manual), laboratory coat, and appropriate gloves must be worn while performing this procedure. Nitrile gloves shall be worn when handling solvents; latex gloves may be worn when handling samples only; and cut resistant gloves shall be worn when washing glassware.

## 5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS 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 SDS for each material before using it for the first time or when there are major changes to the SDS.

Material <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Material <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	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.
Hexane	Flammable	50 ppm (TWA)	Prolonged or repeated contact with skin can cause defatting and dermatitis. Contact with eyes can cause redness, tearing, and blurred vision. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
<p>(1) Always add acid to water to prevent violent reactions.            (2) Exposure limit refers to the OSHA regulatory exposure limit.</p>			

## 6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

### 6.1 Equipment

#### 6.1.1 Microwave extractor. CEM MARS®

At least once a year, power measurement calibration should be performed at 400 W, 800 W, and 1600 W. This calibration can be performed by the vender or by TestAmerica staff following the instructions in the Operations Manual for the microwave.

#### 6.1.2 Microwave extraction vessels. 75 mL Teflon™ Express vessels with stopper and cap (CEM Corp.)

#### 6.1.3 Hand wrench to tighten the caps on the extraction vessels.

#### 6.1.4 MARS 40 position carousel (CEM Corp)

#### 6.1.5 Balance, >1400-g capacity, accurate to ± 0.1 g, calibrated daily per SOP DV-QA-0014.

## 6.2 Supplies

- 6.2.1 Media bottles, 100 mL or 250 mL capped with aluminum foil.
- 6.2.2 Stainless steel conical funnels
- 6.2.3 Ashless cellulose filter paper
- 6.2.4 Pipetter with disposable 1.0-mL tips, calibrated daily per SOP DV-QA-0008.
- 6.2.5 Metal spatulas or tongue depressors.
- 6.2.6 Solvent dispenser pump.
- 6.2.7 Filter flask.
- 6.2.8 Vacuum pump.
- 6.2.9 Washing tool for Teflon™ extractor vessels. This tool is a long thin sponge-like brush.

## 6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

## 7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

- 7.1 Methylene chloride – Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.
- 7.2 Acetone - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.
- 7.3 Hexane - Each lot of solvent is tested following CA-Q-S-001 or CA-Q-S-001-DV-1 before it is put into use. QA personnel post the list of approved lots at solvent

storage areas.

- 7.4 Baked Sodium Sulfate, 12-60 mesh - Heat sodium sulfate in a 400°C oven for at least four hours. QA personnel post the list of approved lots at solvent storage areas.
- 7.5 Baked Ottawa Sand – Heat Ottawa sand in a 400°C oven for at least four hours.
- 7.6 35% Nitric Acid – Dilute concentrated (70%) Nitric Acid 1:1 in water.
- 7.7 Standards - Please reference SOP DV-OP-0020 and WI-DV-009 for information regarding the surrogate and spike standards used in this procedure.

## 8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time <sup>1</sup>	Reference
Soils for Method 8082A <sup>2</sup>	Glass with Teflon-lined lids	30 grams	Cool, ≤ 6°C	None	SW-846
Wipes for Method 8082A <sup>2</sup>	Glass with Teflon-lined lids	N/A	Cool, ≤ 6°C	None	SW-846
Soils for all other Methods, including 8082	Glass with Teflon-lined lids	30 grams	Cool, ≤ 6°C	14 days	SW-846
Wipes for all other Methods, including 8082	Glass with Teflon-lined lids	N/A	Cool, ≤ 6°C	14 days	SW-846

<sup>1</sup> Exclusive of analysis.

<sup>2</sup> Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require the 14 day holding time for Method 8082. The states of Alabama, California, Colorado, Connecticut, Nevada, New Jersey, Pennsylvania, and Rhode Island require the 14 day holding time for Method 8082.

## 9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

- 9.1.2** Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.
- 9.1.3** Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.
- 9.1.4** Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

## **9.2** Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

## **9.3** Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

## **9.4** Method Blank (MB)

- 9.4.1** A method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.
- 9.4.2** The method blank consists of 30 g of baked Ottawa sand free of any of the analyte(s) of interest.

**9.5** Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

- 9.5.1** At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.
- 9.5.2** The LCS consists of 30 g of baked Ottawa sand to which the analyte(s) of interest are added at known concentration.
- 9.5.3** Method AK102 requires LCS and a LCSD for every batch for every spike compound.

**9.6** Matrix Spike/Matrix Spike Duplicate (MS/MSD)

- 9.6.1** One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.
- 9.6.2** If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.
- 9.6.3** DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD must be prepared.

**9.7** Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

**10.0** Procedure

- 10.1** One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.
- 10.2** Any deviations from this procedure identified after the work has been completed must also be documented as a nonconformance, with a cause and corrective action described.

### 10.3 Critical Procedural Considerations

**10.3.1** As stated throughout this SOP, analysts must review the LIMS Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-009).

**10.3.2** Analysts must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other beaker or media bottle than the designated one should be cleaned or disposed of before coming into contact with the sample.

**NOTE:** Rotate glassware; do **not** use specific glassware, equipment or positions for the MB and LCS/LCSD.

### 10.4 Periodic cleaning.

**10.4.1** At least once every four weeks, the extraction vessels must be cleaned using a "Clean Method" on the microwave. The method is under the User Directory with the settings that follow:

- Sample Type: Inorganic
- Control Type: Ramp to Temperature
- Power: 100%
- Ramp: 5 minutes to 180°C
- Hold: 10 minutes

**10.4.2** Fill each tube with 30 mL of the nitric acid solution described in Section 7 and cap tightly. Place the tubes in the carousel, then run the "Clean Method"

**10.4.3** Allow the vessels to cool, and then dispose of the nitric acid in waste stream J. Rinse the vessel with DI water three times.

**10.4.4** Fill each tube with 30 mL of 1:1 Methylene Chloride: Acetone solution and cap tightly. Place the tubes in the carousel, then run the "Clean Method" again.

**10.4.5** Allow the vessels to cool, and then dispose of the solvent in waste stream CA. Allow the vessels to air dry.

### 10.5 Assemble and Clean the Extraction Tubes Immediately Before Use.

**10.5.1** If the microwave tube, cap, or plugs are wet, pre-rinse with acetone.

**10.5.2** Rinse the microwave tube, cap and plug twice with methylene chloride. The plugs can be placed in a large glass jar to help facilitate the rinse.

**10.5.3** Discard the solvent in the correct waste stream.

**10.6** Aliquot Samples

**10.6.1** If the sample is a soil, mix and homogenize samples according to the instructions provided in SOP DV-QA-0023, Subsampling. If the sample is a wipe, transfer the wipe to the extraction vessel.

**10.6.2** Label microwave vessel with the sample ID, method, and batch number. The label needs to be flat.

**10.6.3** Do not use specific vessels or carousel positions for the MB and LCS.

**10.6.4** For each MB and LCS sample, weigh 30 to 33 g of baked Ottawa sand into labeled microwave vessels. Record a nominal weight of 30 g in the initial volume field, but record the actual weight to the nearest 0.1 g in the notes column.

**10.6.5** For each sample and MS/MSD, weigh 30 to 33 g of sample into the labeled microwave vessel. Record the weight to the nearest 0.1 g directly into LIMS or hand record the weight on the benchsheet.

**NOTE:** If the sample matrix appears to be unusual, or especially wet, the 30 g aliquot can be equally divided between two or three separate microwave extraction vessels. The vessels will be extracted independently, but the extracts will be re-combined before concentration. This will prevent the extraction vessels from over-heating and venting if the sample is unusually wet, oily, or bulky (if a 30 g aliquot would fill the tube more than  $\frac{3}{4}$  full). If the sample is split into two or three separate vessels, prepare an NCM.

**NOTE:** Care should be taken to ensure that the top lip of the tube is clean of any sample material or debris so that the plug will fit tightly later.

**10.6.6** Cap the microwave tube tightly with aluminum foil.

**10.6.7** Place the microwave vessel on a cart next to the sample container so that a second analyst can check the labels. This is documented on the Organic Extraction Checklists (See WI-DV-009).

**10.7** Prepare a bottle with a bottle-top dispenser with the appropriate solvent.

**10.7.1** Methylene Chloride is used for soil and wipe samples for the following methods:

- SW-846 8015B
- SW-846 8015C

- SW-846 8015D
- Alaska Methods AK102 and AK103 (AK102\_103)
- Low-Level NDMA (8270D\_SIM\_LL)

**10.7.2** For soil extraction by all other methods, the solvent used is a 1:1 mixture of methylene chloride and acetone.

**10.7.3** For wipe samples by method 8081 and 8082, the solvent used is hexane.

**10.7.4** For wipe samples by method 8270 SIM, the solvent used is a 1:1 mixture of methylene chloride and acetone.

## **10.8** Add Surrogate and Spike Solutions

**NOTE:** The standards should be allowed to come to room temperature before spiking the samples.

**NOTE:** The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-009.

**10.8.1** Only one batch should be surrogated at a time to ensure the correct standards are used and to ensure the solvent is added as soon as possible to the samples. Document the standards and pipette(s) used on the benchsheet.

**10.8.2** Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard (see WI-DV-009) to the microwave vessel for each field sample and QC sample. Do this by punching a hole in the aluminum foil cap with the pipette tip. Record the ID of the standard used on the benchsheet.

**NOTE:** If the sample aliquot was split into two or three separate tubes in Section 10.6.5 above, split the surrogate volume into the separate tubes as well.

**10.8.3** Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard (see DV-OP-009) to the microwave vessel containing any LCS, LCSD, MS, and MSD samples. Do this by punching a hole in the aluminum foil cap with the pipette tip. Record the ID of the standard used on the benchsheet.

**NOTE:** If the MS or MSD aliquot was split into two or three separate tubes in Section 10.6.5 above, split the spike volume into the separate tubes as well.

**10.9** Making sure not to overflow the vessel, remove the foil cap, and slowly add

approximately 25-30 mL of the appropriate solvent to the vessel. See Section 10.7 above for the appropriate solvent. Note that the solvent should be added as soon as possible after the addition of the surrogate and spiking standards to prevent loss of the more volatile compounds.

**NOTE:** For wipe samples add the solvent to the container that the wipe was received in and then transfer it to the microwave vessel. This is done to ensure a quantitative transfer of any solvent and material in the wipe sample container.

**NOTE:** The solvent should completely cover and saturate the sample so additional solvent may be needed depending on the matrix of the individual sample. The sample and solvent must not fill more than 2/3 of the vessel.

**10.10** Seal the vessels by placing the plug on top of the vessel, small side down, and hand tighten the cap over the plug.

**NOTE:** Care should be taken to ensure that the plug, the cap, and the threads of the vessel are clean of any material or debris.

**10.11** After being sealed, the vessels must be inverted several times to ensure that the material is well mixed and saturated. It is recommended that when extracting with 100% methylene chloride to vent and re-cap the vessels before continuing to relieve excess pressure and thereby preventing the vessels from venting during the extraction.

**10.12** Load vessels into the carousel.

**10.12.1** There must be at least 8 vessels in the carousel. Adding blank vessels with sand and solvent may be necessary.

**10.12.2** Balance the tubes around the carousel to ensure that all samples are exposed to an equal amount of energy during the extraction. See Attachment 1 for details. Only samples using the same extraction solvent should be placed in the same carousel and run at the same time.

**10.12.3** For the vessels to be correctly loaded in the carousel the cap should completely touch the top of the carousel with no other part of the extraction vessel visible.

**10.13** Place the carousel into the microwave, making sure that it sits on the turning apparatus correctly. The carousel should be able to rotate. Close the door.

**10.14** The Method Menu screen should indicate "Start Current Method" as being 3546 Full Xpress. Press the green "Start/Pause" button to begin the extraction.

**NOTE:** If a different method is shown, go to the "Load Method" on the menu screen. Choose "User directory" and place the cursor on the desired

method. Press the "Home" button to return to the main menu, where the test highlighted will appear under the "Start Current Method".

**10.14.1** The method is under the User Directory with the settings that follow:

- Sample Type: Organic
- Control Type: Ramp to Temperature
- Power: 100% (1600 W)
- Ramp: 20 minutes to 115°C
- Hold: 10 minutes

**10.14.2** When the extraction is complete, the vessels will need to return to room temperature prior to opening the vessels. The microwave will indicate the approximate temperature of the vessels.

**CAUTION:** If the carousel is removed from the microwave before the vessels are at room temperature, do NOT open the vessels. The vessels may be placed in a rack outside of the microwave to cool down.

**10.14.3** The microwave contains a solvent sensor that will indicate the presence of solvent in the microwave and will stop the extraction. To minimize this, care needs to be taken not to overfill the vessel and to properly cap and tighten the vessel prior to extraction. If the solvent sensor indicates the presence of solvent, open the door and inspect the tops of the tubes for evidence of a solvent leak. If solvent has vented or leaked out of an extraction vessel, the sample must be re-aliquotted and the extraction started over. It is best to re-aliquot the sample into two or three separate extraction vessels to prevent over-heating again. Document this in an NCM.

**10.15** Assemble and Clean Filter Funnels and Media Jars.

**10.15.1** Without gloves on, fold a 18 cm diameter cellulose filter paper in quarters. Open the folds to create a cone. Place the filter paper in the bottom of a conical stainless steel funnel. Place the funnel on a 100 mL or 250 mL media bottle.

**NOTE:** For low-level NDMA samples by method 8270D\_SIM\_LL, use designated glass funnels instead of the stainless steel funnels and instead of re-usable media jars, use disposable amber bottles. This is done to prevent contamination.

**10.15.2** Place approximately 1 tablespoon of baked sodium sulfate in the funnel. Rinse all surfaces of the funnel, the filter and the sodium sulfate with the extraction solvent (see Section 10.7), so all surfaces of the funnel, filter, and sodium sulfate are rinsed.

**NOTE:** When preparing glassware for the extraction of wipe samples, sodium sulfate is not necessary and the solvent used in the rinse should be the solvent used in the extraction of the wipe samples. (Normally hexane for methods 8081 and 8082).

**10.15.3** Allow the solvent to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional solvent to the rinse if necessary.

**10.15.4** Pour the solvent out of the media bottle over the stem of the stainless steel funnel to rinse the funnel stem.

**10.15.5** Discard the solvent in the correct waste stream.

#### **10.16** Filter the Extracts

**10.16.1** After the extraction method is complete and the vessels reach room temperature, quantitatively transfer the entire sample through solvent rinsed sodium sulfate funnels and into the media jar. The quantitative transfer is performed by rinsing the microwave extraction vessel at least three times with solvent.

**NOTE:** The quantitative rinse is vital in order to achieve good recoveries. The rinses should be significant enough that when done, the extract volume is between 75 mL and 100 mL.

**NOTE:** If the sample aliquot was split between two or three tubes, the extracts from all the tubes shall be combined at this time. Filter all of the extracts through the same sodium sulfate funnel and collect in the same media jar.

**10.16.2** Once the solvent has completely drained into the collection apparatus, rinse the funnel contents with 10 to 20 mL of additional solvent. Dispose of the solid sample and sodium sulfate into Waste Stream D and cap the media jar with aluminum foil.

**10.17** If the extract contains visible solids, it will be necessary to filter the extract again prior to concentration.

**10.18** Store the extract refrigerated at  $\leq 6^{\circ}\text{C}$  until concentration. Ensure that the extracts in 1:1 Methylene chloride:acetone are placed in a flammable rated refrigerator.

**10.19** Handwritten notes on the benchsheet are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-009).

**10.20** All glassware and microwave tubes, plugs, and caps are washed according to DV-OP-0004.

## **10.21 Maintenance**

- 10.21.1** As needed, wipe out the inside and outside of the microwave with a damp cloth.
- 10.21.2** See Section 10.4 for vessel cleaning.
- 10.21.3** At least once a year, power measurement calibration should be performed at 400 W, 800 W, and 1600 W. This calibration can be performed by the vender or by TestAmerica staff following the instructions in the Operations Manual for the microwave.

## **10.22 Troubleshooting**

- 10.22.1** If it appears that the solvent sensor is malfunctioning, ensure that the sensor is aligned at a 45 degree upward angle on the back of the unit.
- 10.22.2** The snorkel vent should be set inside of a hood, but care should be taken so that the opening is not blocked. Make sure the snorkel does not press against the back of the hood.

## **11.0 Calibration**

Not applicable to this procedure.

## **12.0 Calculations / Data Reduction**

Not Applicable.

## **13.0 Method Performance**

### **13.1 Method Detection Limit Study (MDL)**

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

### **13.2 Limit of Quantitation Verification (LOQV)**

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

### **13.3 Demonstration of Capabilities**

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

- 13.3.1** Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.
- 13.3.2** Calculate the average recovery and standard deviation of the recovery for each analyte of interest.
- 13.3.3** If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.
- 13.3.4** Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.
- 13.3.5** Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

### **13.4 Training Requirements**

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

### **14.0 Pollution Control**

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

### **15.0 Waste Management**

- 15.1** All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been

implemented to minimize the potential for pollution of the environment. Employees will abide by this method, the policies in section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention", and the Waste Management procedure, DV-HS-001P.

## **15.2 Waste Streams Produced By This Method**

**15.2.1** Methylene chloride – Waste Stream B

**15.2.2** 1:1 MeCl<sub>2</sub>:Acetone – Waste Stream CA

**15.2.3** Flammable solvent – Waste Stream C

**15.2.4** Solid waste/sodium sulfate – Waste Stream D

**15.2.5** Nitric Acid Waste – Waste Stream J

**15.2.6** Expired Standards/Reagents – Contact Waste Coordinator for guidance

**NOTE:** Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

## **16.0 References / Cross-References**

**16.1** SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3456 Microwave Extraction, Revision 0, February 2007.

**16.2** Alaska Method AK102, "For the Determination of Diesel Range Organics", Version 04/08/02.

**16.3** Alaska Method AK103, "For the Determination of Residual Range Organics", Version 04/08/02.

**16.4** NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water", Manchester Environmental Laboratory, Dept of Ecology, State of Washington.

## **17.0 Method Modifications:**

**17.1** SW-846 Method 3546 calls for samples to be either air-dried and ground or mixed with sodium sulfate prior to extraction. This procedure does not call of the air-drying of samples unless requested by the client as this may lead to loss of the more volatile compounds. Sodium sulfate is not used in the extraction vessel, rather the extracts are dried with sodium sulfate after extraction and prior to concentration. Salts are known to superheat when exposed to microwave energy.

- 17.2** SW-846 Method 3546 calls for samples to be aliquoted on a balance capable to weighing to 0.01 g. This SOP calls for a balance capable to weighing to 0.1 g as this is sufficient to report data to 3 significant figures.
- 17.3** SW-846 Method 3546 Section 1.4 states “2-20 g of material is usually necessary and can be accommodated by this extraction procedure.” This SOP calls for 30-33 g of material.
- 17.4** SW-846 Method 3546 Section 11.7 states “Add approximately 25 mL of the appropriate solvent system to the vessel.” This SOP calls for the addition of 25-30 mL of solvent.
- 17.5** Method NWTPH-Dx calls for samples to be extracted by method SW-846 3550C. Valid MDLs and IDOCs have been completed using both method SW-846 3550C and SW-846 3546 and they are comparable therefore method NWTPH-Dx is a possible determinative method by this procedure.
- 17.6** Method AK102 and AK103 calls for samples to be extracted by soxhlet. Valid MDLs and IDOCs have been completed using this procedure, therefore method AK102 and AK103 are listed as a possible determinative methods by this procedure.

## **18.0 Attachments**

Table 1: Determinative Methods Using Microwave Extraction

Attachment 1: Proper Carousel Loading

## **19.0 Revision History**

- Revision 7, January 31, 2017
  - Annual Technical Review
  - Added paragraph to Section 3.0 referencing the QAM for general definitions
  - Added paragraph to Section 6.0 to record IDs of pipettes and equipment
  - Updated language in Section 9.6.3 requiring LCSDs when no MS/MSD
  - Added note to Section 10.3.2 on rotating glassware/equipment/positions
  - Added current Section 13.2 defining LOQV
- Revision 6, January 31, 2016
  - Annual Technical Review
  - Updated Section 9.1 to contain verbiage consistent with other SOPs
  - Added Section 9.6.3 regarding DoD MS/MSD requirements
  - Changed the “Clean Method” frequency from two to four weeks in Section 10.4.1
  - Changed the waste stream from C to CA in Section 10.4.5
  - Section 10.5.2 changed the rinse requirement to be performed twice.
  - Added Section 10.6.3 instructing not to use specific vessels or positions for the MB and LCS.
  - Modified Section 10.6.4 weight recording requirements

- Added Section 10.6.6 – cap with aluminum foil
- Added the documentation of the standards and pipette used in Section 10.8.1
- Clarified the need to punch a hole in foil when spiking to Section 10.8.2 & 10.8.3
- Clarified the process for adding solvent to vessels in Section 10.9
- Added the requirement to place 1:1 Methylene chloride:acetone extracts in a flammable rated refrigerator to Section 10.18
- Revised Section 13.1 – Method Detection Limit Study (MDL)
- Revised Section 13.2 – Demonstration of Capabilities
- Revised Section 13.3 - Training Requirements
- Updated Section 17.4 to reflect the addition of 25-30 mL of solvent
- Archived all revision histories 2010 and earlier
- Revision 5, January 31, 2015
  - Annual Technical Review
  - Reformatted SOP
  - Revised Section 7.4 to remove the requirement to test the sodium sulfate before use. This was done to reflect current practice in CA-Q-S-001-DV-1.
  - Added “NWTPH DRO” to the procedure
  - Revised Section 10.5.2 to state that the plugs and caps can be rinsed in a large glass jar.
  - Added a note in Section 10.15.1 to state that for method 8270D\_SIM\_LL, designated glass funnels and disposable amber bottles will be used to filter the extracts.
  - Added Sections 16.2-16.5 to list AK102, AK103, and NWTPH methods as references.
  - Removed Section 17.8, redundant with 17.5.
  - Updated Table 1 to reference the correct methods and SOPs.
- Revision 4, January 31, 2014
  - Annual Technical Review
  - Revised Section 1.2 to state that the procedure may be used for additional methods when appropriate solvents are used instead of pH as there are no pH adjustments made in the procedure.
  - Removed Teflon™ lined caps from the Equipment and Supplies list in Section 6 as the lab now uses aluminum foil.
  - Added footnote to the table in Section 10 stating some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require 14 day hold time for method 8082A.
  - Revised Section 9.1.2 to state that this procedure meets all criteria of DoD QSM 5.0.
  - Revised Section 9.4 to clarify that one method blank is processed with each batch.
  - Removed “Acceptance Criteria” and “Corrective Action” information from Sections 9.4, 9.5, 9.6, and 9.7. This information can be found in the analytical SOPs.
  - Added a bullet point in Section 10 to clarify that any deviations discovered after the procedure is performed are to be documented in an NCM.
  - Revised Section 10 to remove the instruction to place the label towards the bottom of the vessel. This is not necessary. Also removed the requirement that the label must include the date. The label includes the batch number, which is unique and the date of extraction is recorded in the batch.

- Revised the procedure to state the periodic acid cleaning of the tubes should be done at least once every two weeks instead of weekly.
- Removed methods “NWTPH DRO” and “Okla\_DRO” from the procedure. The lab does not perform microwave extraction for these methods at this time.
- Added sub-sections for Maintenance and Troubleshooting to Section 10 per DoD QSM 5.0.
- Added low-level NDMA and 8015D as a possible analytical method to Section 10 and to Table 1
- Removed 8310 as a possible analytical method in Table 1.
- Added Attachment 1 to give instructions on how to properly load the vessels in the carousel.
- Revision 3, January 31, 2013
  - Annual Technical Review
  - Sections 4.2 and 10.5.4 were revised to remove the optional addition of sodium sulfate to the samples before extraction. It was determined that the better option when dealing with wet samples is to split the sample into two or three tubes and re-combine the extracts before concentration.
  - Section 4 was revised to add instructions on how to deal with paint chip samples.
  - Section 5 was revised to add comments about the dangers of metal fragments in samples.
  - Section 6 was revised to include the requirement that the Power Measurement Calibration procedure be performed on the unit every year.
  - Section 8 was revised to update the hold times for Method SW-846 8082A.
  - Section 10.8 was revised to give more detail on how full the extraction vessel should be once solvent has been added.
  - Section 10.13.1 was revised to allow the carousel to be removed from the microwave unit before the vessels are cool so long as the vessels are not opened.
  - Section 10.15.1 was revised to add a note about the importance of quantitative transfers and rinses while filtering the extracts.
  - Section 10.15.1 was revised to add instructions to combine all extracts from samples that were originally split across two or three tubes.
  - Section 15 was revised to include the waste stream CA.
  - Added the Note to Table 1
- Revision 2.0, January 31, 2012
  - Annual Technical Review
  - Updated Section 4.2 and Section 10.5.4 to describe when sodium sulfate should be used in the extraction vessel.
  - Updated Section 6.0 to allow the use of aluminum foil to cap 100mL and 250mL media jars.
  - Updated Section 6.1 to include details on computer software and hardware.
  - Updated Section 7.0 to include details on the purity of reagents and standards.
  - Updated Section 9.1.4 and Section 10.1 to more accurately reflect the NCM process.
  - Corrected grammatical and formatting errors
  - Updated Section 10.3 to include a solvent cleaning after the weekly acid cleaning.
  - Updated Section 10.5.4, Section 10.7.2, and Section 10.7.3 to include an option to split the sample aliquot into two separate microwave vessels.

- Updated Section 10.10 and 10.13.2 to give details on how to prevent vessels from over-heating and venting and steps to be taken if venting does occur.
- Updated Section 10.16 to accurately reflect how the laboratory handles extracts with suspended sediment.
- Updated Section 10.19 to reference SOP DV-OP-0004 on how to clean the microwave vessels.
- Revision 1 dated 01 Jan 2011
  - Added 8270C SIM as a valid determinative method by microwave extraction.
  - Changed the procedure to call for the extract to be filtered thru a conical steel funnel lined with cellulose filter paper instead of a glass funnel with glass wool. This was done to help remove sediment from the extracts.
  - Removed details about the surrogate and spike standards used in the extraction. This information can now be found in DV-OP-0020.
  - Added instructions to Section 7 on how to prepare the nitric acid solution used in the weekly cleaning of the tubes.
  - Changed the solvent used in the extraction of samples for method 8081 and 8082. The samples are now extracted in a 1:1 Mixture of MeCl<sub>2</sub>:Acetone instead of a 1:1 Mixture of MeCl<sub>2</sub>:Hexane.
  - Revised the procedure in Section 10.5 for aliquotting samples to state that 30 to 33g of sample should be used instead of 30±2g and that the weight should be recorded to the nearest 0.1g instead of the nearest mg.

*Earlier revision histories have been archived and are available upon request.*

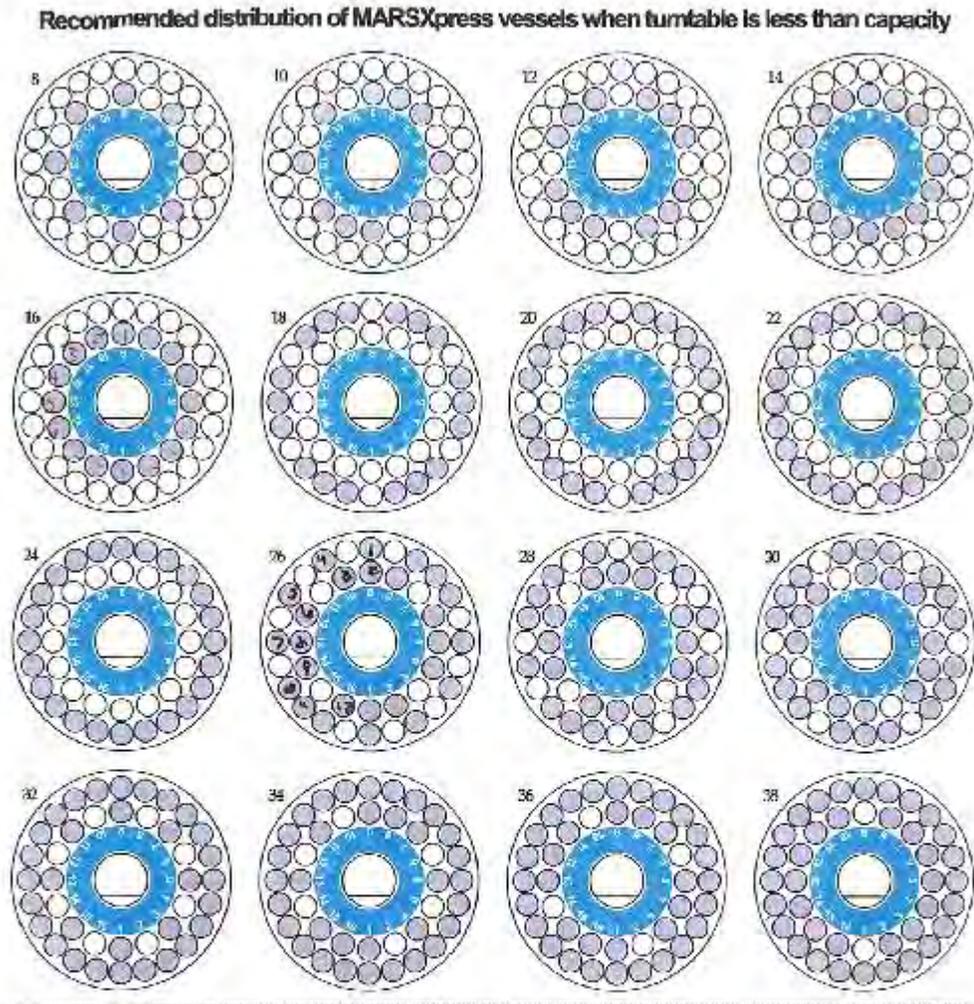
**TABLE 1.**

**Determinative Methods Using Microwave Extraction**

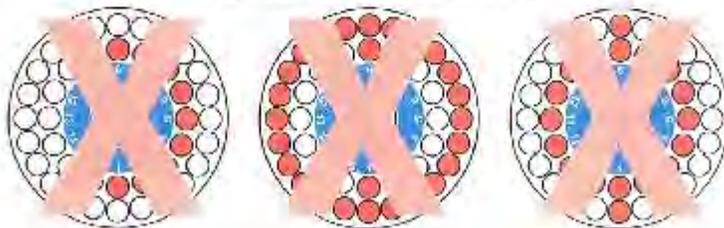
Method Description	Determinative Method	SOP
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls (PCBs)	SW-846 8082 SW-846 8082A	DV-GC-0021
Diesel and Residual Range Organics	SW-846 8015B SW-846 8015C SW-846 8015D NWTPH-Dx AK102 AK103	DV-GC-0027
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM SW-846 8270D SIM	DV-MS-0002
Low-Level NDMA by Isotope Dilution, GC/MS SIM, Large Volume Injection	SW-846 8270C/D SIM	DV-MS-0015

## ATTACHMENT 1.

### Proper Carousel Loading

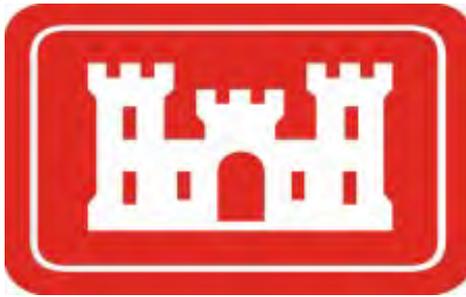


**Incorrect distribution: What not to do**



## **APPENDIX C**

### **Site Signage during Remedial Activities**



# Remedial Action for the Former U.S. Border Protection Firing Range (Nogales, AZ)

**Site Name and Location:** Former U.S. Border Protection Firing Range,  
1651 W. Target Range Road, Nogales, Arizona

## **Summary of Planned Work:**

The United States Army Corps of Engineers (USACE) has retained the Joint Venture formed by Sol Solutions, LLC and J.C. Palomar (Sol-JCP) to provide a Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) Remedial Action for the removal of contaminated soil at the former U.S. Customs and Border Protection (USCBP) Firing Range.

## **Contact Information:**

USACE: David Clark (817-886-1876)

USCBP: Joseph Zidron (949-643-6392)

Arizona Department of Environmental Quality: Nicole Osuch (602-771-4847)

Sol-JCP: Robert Noyes (480-544-7045)

**APPENDIX D**

**Backfill Information Letter from CalPortland Plant 101**



April 23, 2018

JC Palmar

Project: Nogales BP Firing Range  
Nogales, Arizona

**RE: Bedding Sand Commodity Code - 90000345**

Ladies and Gentlemen:

Please find attached the referenced material submittal as requested.

This material will be produced at CalPortland Plant 101 located at 409 Camino Ramanote Rio Rico Arizona 85648. The material mined from this location is virgin material. This is a commercial aggregate source CM 0426 that has been in operation for the past 50 years.

Should you have any questions regarding this submittal, please call.

Sincerely,

**CalPortland**

A handwritten signature in blue ink that reads 'Tom Romero'.

Tom Romero  
Quality Control Director



**Western Technologies Inc.**  
The Quality People  
Since 1955

3480 South Dodge Boulevard  
Tucson, AZ 85713  
(520) 748-2262

**PHYSICAL PROPERTIES OF SOILS & AGGREGATES**

Client **CALPORTLAND**  
**6601 N. CASA GRANDE HWY**  
**TUCSON, AZ 85743**

Date of Report **09-14-17**  
Job No. **2947JG029**  
Event / Invoice No. **C029-020**  
Authorized by **TOM ROMERO**  
Sampled by **S. PRIES**  
Submitted by **S. PRIES**  
Source / Location Designated by **S. PRIES**

Lab No. **4843**  
Date **09-09-17**  
Date **09-09-17**  
Date **09-09-17**  
Date **09-09-17**

Project **ANNUAL QUALITY CONTROL**  
Location **TUCSON, AZ**  
Type / Use of Material **BEDDING SAND/VARIOUS**  
Supplier / Source **CALPORTLAND/RIO RICO (101R)**  
Sample Source / Location **STOCKPILE/PLANT**  
Special Instructions

**TEST RESULTS**

SIEVE ANALYSIS : AZ 201 FINER THAN NO. 200 :			LABORATORY COMPACTION CHARACTERISTICS :		METHOD		
SIEVE	ACCUMULATIVE % PASSING	SPECIFICATION			SAMPLE PREPARATION: <input type="checkbox"/> WET <input type="checkbox"/> DRY RAMMER USED: <input type="checkbox"/> 2 IN. CIRCULAR FACE <input type="checkbox"/> OTHER <input type="checkbox"/> MECHANICAL <input type="checkbox"/> MANUAL  MAXIMUM DRY UNIT WEIGHT, LBF/FT <sup>3</sup> → OPTIMUM WATER CONTENT, % →  OVERSIZE AGGREGATE : BULK SPECIFIC GRAVITY : ABSORPTION, % : % OVERSIZE IN LAB SAMPLE :  SPECIFIC GRAVITY IN ZERO AIR VOID CURVE :		
6"							
4"							
3"							
2"							
1 1/2"							
1 1/4"							
1"							
3/4"							
1/2"							
3/8"							
1/4"							
No.4	<b>100</b>						
8	<b>16</b>						
10	<b>9</b>						
16	<b>4</b>						
30	<b>3</b>						
40	<b>2</b>						
50	<b>2</b>						
100	<b>2</b>						
200	<b>1.5</b>						
			WATER CONTENT, % DRY WEIGHT				
TEST PROCEDURE			RESULT	SPECS	TEST PROCEDURE	RESULT	SPECS
<b>LIQUID &amp; PLASTIC PROPERTIES AASHTO T89, 90</b> METHOD B ESTIMATED % RETAINED ON NO. 40 <b>0</b> LIQUID LIMIT → SAMPLE AIR DRIED <input checked="" type="checkbox"/> YES <input type="checkbox"/> NO PLASTIC LIMIT → PLASTICITY INDEX →			<b>NP</b>		<b>RESISTANCE TO DEGRADATION OF SMALL-SIZE COARSE AGGREGATES BY ABRASION :</b> GRADING 100 REV, % LOSS → GRADING 500 REV, % LOSS →		
<b>MOISTURE CONTENT :</b> PORTION TESTED % DRY WEIGHT →					<b>SPECIFIC GRAVITY :</b> MAX. PARTICLE SIZE, IN. SPECIFIC GRAVITY @ 20°C →		
<b>EXPANSION / COMPRESSION PROPERTIES OF COHESIVE SOIL :</b> <input type="checkbox"/> EXPANSION <input type="checkbox"/> COMPRESSION, % → SURCHARGE, PSF MAXIMUM SWELL PRESSURE, KSF →					<b>pH DETERMINATION : AZ 236</b> pH → <b>9.2</b>		
INITIAL WATER CONTENT, % DRY UNIT WEIGHT LBF/FT <sup>3</sup>					<b>SOLUBLE SALTS :</b> PPM →		
<b>EXPANSION INDEX OF SOIL :</b> INITIAL WATER CONTENT, % : INITIAL DRY UNIT WEIGHT LBF/FT <sup>3</sup> : INITIAL DEGREE OF SATURATION : FINAL WATER CONTENT, % :			<b>EI →</b>		<b>MINIMUM RESISTIVITY : AZ 236</b> OHM-CM → <b>11033</b>		
					<b>SOIL CLASSIFICATION :</b> NAME:	<b>GROUP SYMBOL:</b>	

Comments :

Copies to : **CLIENT**

THE SERVICES REFERRED TO HEREIN WERE PERFORMED IN ACCORDANCE WITH THE STANDARD OF CARE PRACTICED LOCALLY FOR THE REFERENCED METHOD(S) AND RELATE ONLY TO THE CONDITION(S) OR SAMPLE(S) TESTED AS STATED HEREIN. WESTERN TECHNOLOGIES INC. MAKES NO OTHER WARRANTY OR REPRESENTATION, EXPRESSED OR IMPLIED, AND HAS NOT CONFIRMED INFORMATION INCLUDING SOURCE OF MATERIALS SUBMITTED BY OTHERS.

REVIEWED BY \_\_\_\_\_

(SIGNED COPY ON FILE)