SESCO group
ENVIRONMENTAL SOLUTIONS

QUALITY ASSURANCE PROJECT PLAN

Dixon Road Site
Kokomo, Indiana

Prepared for:
United States Environmental Protection Agency
77 West Jackson Blvd
Chicago, IL 60604

By:
SESCO Group
1426 W. 29th Street
Indianapolis, IN 46208

Based on the Intergovernmental Data Quality Task Force Uniform Federal Policy for Quality Assurance Project Plans
(Final Version 1.1, June 2006)

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Attachment B Standard Operating Procedures/Practices
Attachment C Exhibits of Quality Control Logs and Report Forms

INTRODUCTION

The United States Environmental Protection Agency (EPA) requires that all parties involved with environmental monitoring and measurement efforts mandated or supported by the EPA participate in a centrally managed quality assurance program. Any party generating data under this program has the responsibility to implement minimum procedures to assure that the precision, accuracy, completeness, comparability, and representativeness of its data is known and documented. To ensure that this responsibility is met, each party must prepare a written plan covering the project(s) it is to perform.

This Quality Assurance Project Plan (QAPP) has been prepared by SESCO Group (SESCO). This QAPP has been prepared in accordance with EPA Uniform Federal Policy (UFP) for QAPP guidance and contains information regarding functional activities and specific quality assurance (QA) and quality control (QC) procedures in support of site activities associated with the investigation of the Dixon Road Site (Site). This QAPP also describes the specific protocols, which will be followed for sampling, sample handling and storage, chain-of-custody, and laboratory (and field) analysis.

The Site historically operated as a landfill, auto parts sales, towing service, and a metal recycling facility. Reportedly, ash from the adjacent Kokomo Dump Site (EPA Site ID C564) was disposed at the Site. The contaminants of concern (COCs) associated with the Site are volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), metals, polychlorinated biphenyls (PCBs) and dioxins. Leaking drums were discovered by the Indiana Department of Environmental Management (IDEM) in April 2011 exposed in a creek bank at the adjacent Kokomo Dump Site. IDEM documented that drums and waste piles extended onto the Dixon Road Site. The EPA, IDEM, and the Superfund Technical Assessment and Response Team (START) contractor conducted a Site Assessment on December 3, 2012. During the assessment, EPA documented numerous drums and waste piles and waste extending down the bank of Wildcat Creek. Site Assessment activities included sampling surface soil, subsurface soil, waste piles, and buried waste. Results of the USEPA investigation indicated exceedances of IDEM Direct Contact Industrial Screening Levels (DCISLs) for PCB Aroclors 1248 and 1254, lead, mercury, and hexavalent chromium in surface soil samples. Subsurface samples indicated levels of PCB Aroclors 1248 and 1254, arsenic, lead, and mercury above DCISLs. Additionally, lead and cadmium were encountered above the Toxicity Characteristic Leaching Procedure (TCLP) limit in a surface sample, and cadmium was encountered above the TCLP limit in one subsurface sample.

Identified in this QAPP are various laboratory QA/QC requirements that must be met during environmental data generation activities at the site. As needed, additional information will be added to this QAPP. This site specific QAPP is similar in format to the QAPP workbook (Final, March 2005, Part 2A of the UFP-QAPP) provided under EPA's UFP QAPP guidance documents. This site-specific QAPP contains laboratory contact information, accreditation and certifications, the laboratory's current QAPP or QA/QC Plan, laboratory standard operating procedures (SOPs), and information addressing analytical measurements and relating the laboratory reporting limits to the project data quality objectives (DQOs) and the specified State cleanup programs. The QAPP also contains further details regarding the scope of the field investigation, information and DQOs to be followed during sampling, field equipment and use, field and laboratory analyses, and procedures for investigation-derived waste (IDW) management.

The site-specific QAPP will function in conjunction with the Site Health and Safety Plan (HASP), which provides information regarding potential site-specific hazards and the protective measures to be taken and equipment to be used to ensure worker safety and health.

All QA/QC procedures performed by the contractor under this contract will follow applicable professional technical standards, EPA requirements, government regulations and guidelines, and specific project goals and requirements.

QAPP Worksheet #1 – Title and Approval Page

Site Name/Project Name: **Dixon Road Site**
Site Location: **Kokomo, Indiana**

Document Title: Quality Assurance Project Plan for the Dixon Road Site

Lead Organization: U.S. EPA Region 5

Preparer's Name and Organizational Affiliation: Bill Pickard, Sr. Project Manager, SESCO

Preparer's Address, Telephone Number, and E-mail Address: 1426 West 29th Street
Indianapolis, IN 46208
317-347-9590
bpickard@sescogroup.com

Preparation Date (Day/Month/Year): 16 June 2015

Investigative Organization's Project Manager/Date: Brad Adams 7/2/15
Signature

Printed Name/Organization: Brad Adams/Sesco Group

Investigative Organization's Project QA Officer/Date: Brent Graves 7/2/15
Signature

Printed Name/Organization: Brent Graves/Sesco Group

Lead Organization's Project Manager/Date: _____
Signature

Printed Name/Organization: Shelly Lam/U.S. EPA On-Scene Coordinator

Approval Signatures/Date: _____
Signature

Printed Name/Title: Ida Levin/QAPP Reviewer

Approval Authority: U.S. EPA Region 5

Other Approval Signatures/Date: Bill Pickard / 7/2/2015
Signature

Printed Name/Title: Bill Pickard/Senior Project Manager, Sesco Group

Document Control Numbering System : SESCO-DRS-UFPQAPP-003

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All QA/QC procedures performed by the contractor under this contract will follow applicable professional technical standards, EPA requirements, government regulations and guidelines, and specific project goals and requirements.

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Site Location: **Kokomo, Indiana**

Document Title: Quality Assurance Project Plan for the Dixon Road Site

Lead Organization: U.S. EPA Region 5

Preparer's Name and Organizational Affiliation: Bill Pickard, Sr. Project Manager, SESCO

Preparer's Address, Telephone Number, and E-mail Address: 1426 West 29th Street
Indianapolis, IN 46208
317-347-9590
bpickard@sescogroup.com

Preparation Date (Day/Month/Year): 16 June 2015

Investigative Organization's Project Manager/Date: Brad Adams 7/2/15
Signature

Printed Name/Organization: Brad Adams/Sesco Group
Investigative Organization's Project QA Officer/Date: Brent Graves 7/2/15
Signature

Printed Name/Organization: Brent Graves/Sesco Group

Lead Organization's Project Manager/Date: Shelly Lam 7/20/15
Signature

Printed Name/Organization: Shelly Lam/U.S. EPA On-Scene Coordinator

Approval Signatures/Date: Ida Levin 7/16/15
Signature

Printed Name/Title: Ida Levin/QAPP Reviewer

Approval Authority: U.S. EPA Region 5

Other Approval Signatures/Date: Bill Pickard 7/2/2015
Signature

Printed Name/Title: Bill Pickard/Senior Project Manager, Sesco Group

Document Control Numbering System : SESCO-DRS-UFPQAPP-003
4276 Dixon Road QAPP 003

QAPP Worksheet #2 – QAPP Identifying Information

Site Name/Project Name: Dixon Road Site

Site Location: Kokomo, Indiana **Revision Number: 1.0**

Site Number/Code: C5M5 **Revision Date:** N/A

Operable Unit: Not Applicable

Contractor Name: SESCO Group

Contractor Number: NA

Contract Title: NA

Work Assignment Number: NA

1. Identify regulatory program: U.S. EPA Region 5, CERCLA,
2. Identify approval entity: U.S. EPA Region 5
3. The QAPP is (select one): ☐Generic ☒Project Specific
4. List dates of scoping sessions that were held: NA
5. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
Not Applicable	

6. List organizational partners (stakeholders) and connection with lead organization:

   EPA Region 5

7. List data users:

   OSCs, Regional Counsel, PRPs, Region 5 contractors, state, and local officials performing work for Region 5, etc.

8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

QAPP Worksheet #2 – QAPP Identifying Information (continued)

UFP-QAPP Worksheet #	Required Information	Cross-Reference to Related Information
A. Project Management		
	Documentation	
1	Title and Approval Page	2
2	QAPP Identifying Information	3
3	Distribution List	6
4	Project Personnel Sign-Off Sheet	7
	Project Organization	
5	Project Organizational Chart	8
6	Communication Pathways	9
7	Personnel Responsibilities and Qualifications Table	10
8	Special Personnel Training Requirements Table	11
	Project Planning/ Problem Definition	
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	12
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	13
11	Site-Specific Project Quality Objectives	15
12	Measurement Performance Criteria Table	17
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	30
14	Summary of Project Tasks	31
15	Reference Limits and Evaluation Table	33
16	Project Schedule/Timeline Table	49
B. Measurement Data Acquisition		
	Sampling Tasks	
17	Sampling Design and Rationale	51
18	Sampling Locations and Methods/ SOP Requirements Table Sample Location Map(s)	52
19	Analytical Methods/SOP Requirements Table	54
20	Field Quality Control Sample Summary Table	60
21	Project Sampling SOP References Table Sampling SOPs	62
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	63
	Analytical Tasks	
23	Analytical SOPs Analytical SOP References Table	64
24	Analytical Instrument Calibration Table	65
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	66
	Sample Collection	

UFP-QAPP Worksheet #	Required Information	Cross-Reference to Related Information
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	67
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	68
	Quality Control Samples	
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	71
	Data Management Tasks	
29	Project Documents and Records Table	82
30	Analytical Services Table Analytical and Data Management SOPs	83
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	84
32	Assessment Findings and Corrective Action Responses Table	85
33	QA Management Reports Table	86
D. Data Review		
34	Verification (Step I) Process Table	87
35	Validation (Steps IIa and IIb) Process Table	88
36	Validation (Steps IIa and IIb) Summary Table	90
37	Usability Assessment	92

QAPP Worksheet #3 – Distribution List

List those entities to which copies of the approved QAPP, subsequent QAPP revisions, addenda, and amendments will be sent.

☐ Worksheet Not Applicable (State Reason)

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Shelly Lam	On-Scene Coordinator (OSC)	EPA	(317) 308-3073		lam.shelly@epa.gov	
Ida Levin	Quality Coordinator	EPA	(312) 886-6254		Levin.Ida@epa.gov	
Maria Gonzalez	Attorney	EPA	(312) 866-6630		gonzalez.maria@epa.gov	
Bill Pickard	Senior Project Manager	SESCO	(317) 347-9590		bpickard@sescogroup.com	
Brad Adams	Project Manager	SESCO	(317) 347-9590		badams@sescogroup.com	
Brent Graves	Quality Assurance Manager	SESCO	(317) 908-4645		bgraves@sescogroup.com	
Beth Garrison	Attorney	City of Kokomo, IN	(765) 456-7442		bgarrison@cityofkokomo.org	
David Guevara	Attorney	Taft, Stettinius & Hollister	(317) 713-3453		dguevara@taftlaw.com	
Vernon Graves	Property Owner	None	(765) 438-0802		None	
John Moriarty	Attorney	Plews Shadley Racher & Braun	(317) 637-0700		jmoriarty@psrb.com	

QAPP Worksheet #4 – Project Personnel Sign-Off Sheet

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable sections of the QAPP and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

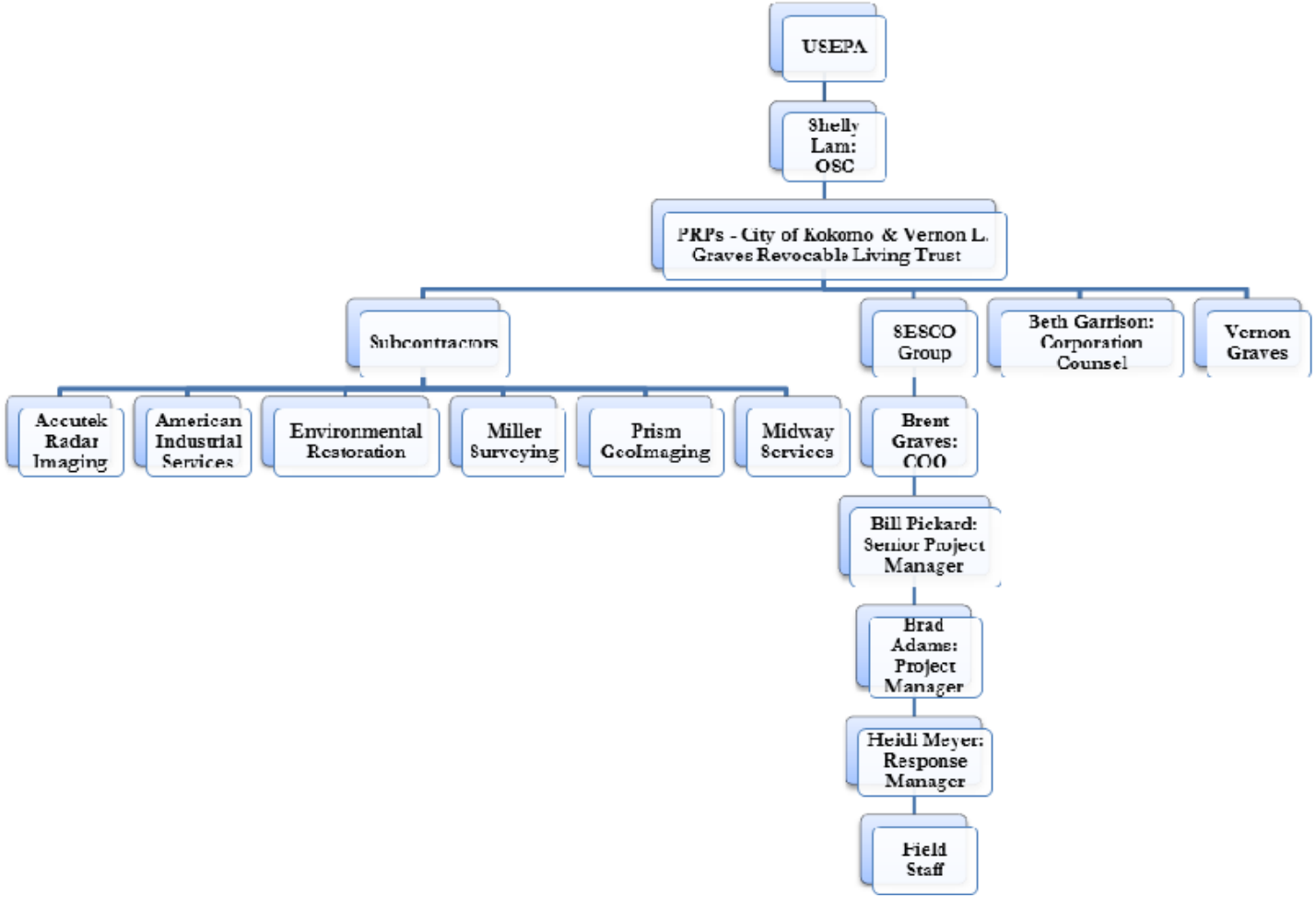
☐ Worksheet Not Applicable (State Reason)

QAPP Recipients	Title	Organization	Telephone Number	Signature
Shelly Lam	On-Scene Coordinator (OSC)	EPA	(317) 308-3073	
Ida Levin	Quality Coordinator	EPA		
Bill Pickard	Senior Project Manager	SESCO	(317) 347-9590	
Brad Adams	Project Manager	SESCO	(317) 347-9590	
Brent Graves	Quality Assurance Manager	SESCO	(317) 908-4645	

QAPP Worksheet #5 – Project Organizational Chart

Identify reporting relationships between all organizations involved in the project, including the lead organization and all contractor and subcontractor organizations. Identify the organizations providing field sampling, on-site and off-site analysis, and data review services, including the names and telephone numbers of all project managers, project team members, and/or project contacts for each organization.

☐ Worksheet Not Applicable (State Reason)



QAPP Worksheet #6 – Communication Pathways

Describe the communication pathways and modes of communication that will be used during the project, after the QAPP has been approved. Describe the procedures for soliciting and/or obtaining approval between project personnel, between different contractors, and between samplers and laboratory staff. Describe the procedure that will be followed when any project activity originally documented in an approved QAPP requires real-time modifications to achieve project goals or a QAPP amendment is required. Describe the procedures for stopping work and identify who is responsible.

☐ Worksheet Not Applicable (State Reason)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
QAPP changes in the field	SESCO Senior Project Manager	Bill Pickard	317-347-9590	Notify the OSC by phone and email of changes to QAPP made in the field and reasons within 2 business days.
QAPP Amendments	SESCO COO/QA Manager	Brent Graves	317-908-4645	Any major changes to the QAPP must be approved by the OSC and EPA QA/QC Manager before the changes can be implemented.
Field corrective actions	SESCO Project Manager	Brad Adams	317-347-9590	The need for corrective action for field issues will be determined by FTL, PM, OSC.
Analytical corrective actions	Laboratory Project Chemist	TBD		The need for corrective action for analytical issues will be determined by laboratory QAO.
Release of analytical data	Laboratory Project Chemist	TBD		No analytical data can be released until verification is completed and laboratory QAO has approved the release.

QAPP Worksheet #7 – Personnel Responsibilities and Qualifications Table

Identify project personnel associated with each organization, contractor, and subcontractor participating in responsible roles. Include data users, decision-makers, project managers, QA officers, project contacts for organizations involved in the project, project health and safety officers, geotechnical engineers and hydrogeologists, field operation personnel, analytical services, and data reviewers. Identify project team members with an asterisk (*). Attach resume to this worksheet or note the location of the resumes.

☐ Worksheet Not Applicable (State Reason)

Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Ida Levin	QAPP Reviewer	U.S. EPA Region 5	Reviews QAPP	
Shelly Lam	OSC	U.S. EPA Region 5	QA oversight	
Brent Graves	Chief Operating Officer	SESCO	Oversees project and responds to EPA	
Bill Pickard	Senior Project Manager	SESCO	Performs QA oversight	
Bradley Adams	Project Manager	SESCO	Manages project – coordinates between lead agency and subcontractor	
Heidi Meyer	Response Manager	SESCO	Oversees H&S for field activities	
TBD	Sampling Team Leader	SESCO	Supervises field sampling and coordinates all field activities	
TBD	Sampling Team Member	SESCO	Performs field sampling and all field activities	
TBD	Data Reviewer	SESCO	Performs data validation	
TBD	Laboratory Manager	Pace Analytical	Manages generation of analytical data	
TBD	Lab QAO	Pace Analytical	Performs lab QA oversight	

QAPP Worksheet #8 – Special Personnel Training Requirements Table

Provide the following information for those projects requiring personnel with specialized training. Attach training records and/or certificates to the QAPP or note their location.

☐ Worksheet Not Applicable (State Reason)

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Project Activities oversight	HAZWOPER and Annual 8-hour Refresher	Tank Industry Consultants	1/31/15	Bradley Adams	Project Manager/SESCO	SESCO Office
Health and Safety oversight	HAZWOPER Supervisor Training	Safety Unlimited, Inc.	10/28/13	Heidi Meyer	Response Manager/SESCO	SESCO Office
Health and Safety oversight	CPR & First Aid Training	American Red Cross	1/2/14	Heidi Meyer	Response Manager/SESCO	SESCO Office
QA Oversight	Uniform Federal Policy for Quality Assurance Project Plans			Brent Graves	QA Manager/SESCO	SESCO Office
Sampling Operations	Health & Safety, HAZWOPER & Annual 8-hour Refresher			TBD	Sampler/SESCO	SESCO Office

QAPP Worksheet #9 – Project Scoping Session Participants Sheet

Complete this worksheet for each project scoping session held. Identify project team members who are responsible for planning the project.

☐ Worksheet Not Applicable (State Reason)

Project Name: Dixon Road Site **Projected Date(s) of Sampling:** TBD **Project Manager:** Brad Adams	**Site Name:** Dixon Road Site **Site Location:** Kokomo, IN

Date of Session: February 27, 2015

Scoping Session Purpose: Define Scope, extent of contamination determination and Sampling requirements

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Bradley Adams	Project Manager	SESCO	(317) 347-9590	badams@sescogroup.com	Project Oversight
Shelly Lam	OSC	U.S. EPA	(317) 417-0980	Lam.Shelly@EPA.gov	Overall Project oversight/AOC Compliance determination
Brent Graves	QA Manager	SESCO	(317) 908-4654	bgraves@sescogroup.com	Project oversight and EPA contact point
Bill Pickard	Senior Project Manager	SESCO	(317) 347-9590	bpickard@sescogroup.com	QA oversight

Comments/Decisions:

Action Items:

Consensus Decisions:

QAPP Worksheet #10 – Problem Definition

Clearly define the problem and the environmental questions that should be answered for the current investigation and develop the project Decision "If…, then…" statements in the QAPP, linking data results with possible actions. The prompts below are meant to help the project team define the problem. They are not comprehensive.

☐ Worksheet Not Applicable (State Reason)

The problem to be addressed by the project:

1. Determine if there are buried drums on the Site and if present, map the area and define the extent of buried drums and contamination in soil, groundwater, sediment, and surface water.
2. Develop and implement a plan to control, contain, and/or remove currently identified drums and any discovered drums & contaminated soil, groundwater, sediment, and surface water.
3. Perform sampling and analysis to determine disposal options

The environmental questions being asked:

1. Does the geophysical survey (GPR, etc.) reliably identify buried drums?
2. If yes, do these drums contain hazardous materials?
3. Are these drums, leaking?
4. Is the surrounding soil and off-site soil contaminated? And to what lateral and vertical extent?
5. Is the on-site and off-site groundwater contaminated? And to what lateral and vertical extent?
6. Has contamination affected Wildcat Creek?
7. Will the sampling to be conducted provide reliable and sufficient data to properly address disposal of the waste?

Observations from any site reconnaissance reports: Leaking drums were discovered by the Indiana Department of Environmental Management (IDEM) in April 2011 exposed in a creek bank at the adjacent Kokomo Dump Site. IDEM documented that drums and waste piles extended onto the Dixon Road Site. The EPA, IDEM, and the Superfund Technical Assessment and Response Team (START) contractor conducted a Site Assessment on December 3, 2012. During the assessment, EPA documented numerous drums and waste piles and waste extending down the bank of Wildcat Creek. Site Assessment activities included sampling surface soil, subsurface soil, waste piles, and buried waste. Results of the USEPA investigation indicated exceedances of IDEM Direct Contact Industrial Screening Levels (DCISLs) for PCB Aroclors 1248 and 1254, lead, mercury, and hexavalent chromium in surface soil samples. Subsurface samples indicated levels of PCB Aroclors 1248 and 1254, arsenic, lead, and mercury above DCISLs. Additionally, lead and cadmium were encountered above the Toxicity Characteristic Leaching Procedure (TCLP) limit in a surface sample, and cadmium was encountered above the TCLP limit in one subsurface sample.

The Site is accessible from the north and east sides of the property, where it is not fenced. Trespassers can potentially be exposed to the high levels of lead and PCB contamination present at the Site. Thus, conditions exist at the Site that support a removal action be conducted to abate threats to human health and the environment.

A synopsis of secondary data or information from site reports: Abandoned drum and contamination was observed on adjacent property during the site assessment by U.S. EPA; contamination and buried waste was observed on subject property during subsequent site assessment by U.S. EPA.

The possible classes of contaminants and the affected matrices: PCBs, VOCs, SVOCs, metals, and dioxins.

The rationale for inclusion of chemical and nonchemical analyses: Historical analytical results and the nature of site operations

Information concerning various environmental indicators:

Project decision conditions ("If..., then..." statements):

1. If buried drums are discovered during the geophysical surveys, limited soil excavation activities will occur to expose these drums and samples will be collected from these drums to identify their waste characteristics
2. If limited excavation is conducted, affected soil will be sampled and if feasible, the leaking drums will be removed, over-packed and staged for proper sampling and disposal.
3. If sampling results from excavation, surface samples, or subsurface soil borings indicate hazardous waste or contaminated soil, groundwater, sediment, or surface water, then a removal action will be conducted at the site to eliminate threats to human health and/or environment both at on-site and off-site locations.

QAPP Worksheet #11- Project Quality Objectives (PQOs)/Systematic Planning Process Statements

Use this worksheet to develop project quality objectives (PQOs) in terms of type, quantity, and quality of data determined using a systematic planning process. Provide a detailed discussion of PQOs in the QAPP. List PQOs in the form of qualitative and quantitative statements. These statements should answer questions such as those listed below. These questions are examples only, however; they are neither inclusive nor appropriate for all projects.

☐ Worksheet Not Applicable (State Reason)

Who will use the data?
Data will be used by SESCO and U.S. EPA OSC.

What will the data be used for?
Physical data will be used by SESCO and U.S. EPA OSC to determine if drums are buried on-site and to define the extent of extent of these buried drums and soil, groundwater, sediment, and surface water contamination at the site. Chemical data will be used to determine hazardous characteristics of the waste and of the soil and groundwater. Project action levels will be derived from the following: TCLP Criteria, TSCA Levels, EPA RMLs, ATSDR data, other federal regulations, and IDEM RSLs for residential and commercial/industrial scenarios.

What types of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?
Analytical data from surface soil, subsurface soil matrices, and solid/liquid matrices data from drums to assess the extent and concentrations of contaminants and migrations; analytical data from potential groundwater (if encountered) to determine risk of vapor intrusion of volatile compounds and potential for migration to Wildcat Creek; geophysical survey to determine if additional drums are present on site; samples to determine disposal methods; samples of media collected after remediation to verify clean up. Analytical groups include metals, VOCs, SVOCs, PCBs, and dioxins. Field screening will be conducted with a VOC detector such as Multi-RAE or similar equipment and XRF to screen samples for metals and select most contaminated samples. Samples will be collected following referenced SOPs and will be analyzed at an off-site commercial laboratory.

How "good" do the data need to be in order to support the environmental decision?
For disposal or confirmation samples, the TSCA levels of: 50 ppm total PCBs or TCLP levels listed under 40 CFR 262 will have to be met. The quantitation limits for all analyses are specified on Worksheet #15. Worksheets #12 and #28 show the measurement performance criteria needed for the quality indicators. Worksheet #20 shows the quality control (QC) samples required for definitive data. All data should pass the data validation tests for use in the reports.

How much data are needed (number of samples for each analytical group, matrix, and concentration)?
Analytical data from 45 surface soil samples, 45 subsurface soil borings (up to 90 samples), drum samples (number TBD in field), and groundwater samples (up to 12).

QAPP Worksheet #11- Project Quality Objectives (PQOs)/Systematic Planning Process Statements (Cont.)

Where, when, and how should the data be collected/generated?
Data will be collected during investigation and waste removal activities.

Who will collect and generate the data?
SESCO and their subcontractors will collect and generate the data.

How will data be reported?
Data can be submitted electronically, by fax, or U.S. Mail to SESCO by the laboratory and other subcontractors.

How will the data be archived?
The chemical data will be archived electronically by SESCO and hard copies of the data will be stored at SESCO's and the City of Kokomo offices

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQIs), measurement performance criteria (MPC), and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for a specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

☐ Worksheet Not Applicable (State Reason)

Matrix	Soil
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
SESCO SOP #WP-01, #WP-04	SW 846 Method 6020 / S-IN-O-031 Mercury 7471A / S-IN-I-040-rev.13 Cr6+ 7196A / S-IN-I-063-rev.11	Precision (field)	RPD ±20%	Field Duplicate	S & A
		Accuracy/Bias (field) Contamination	No analyte > MRL	Equipment Blank	S & A
		Precision (laboratory)	PACE QC Limits	MS/MSD or Sample and Duplicate	A
		Bias (laboratory)	PACE QC Limits	Matrix Spike	A
		Bias (laboratory)	PACE QC Limits	Laboratory Control Sample (LCS)	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*****Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Soil
Analytical Group	Volatile Organics
Concentration Level	Low to medium (µg/kg to mg/kg)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SESCO SOP #WP-01, #WP-04	SW 846 Method 8260/5035 / S-IN-O-029-rev.17	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > RQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
***Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Soil
Analytical Group	Semi-Volatile Organics
Concentration Level	Low to medium (µg/kg to mg/kg)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SESCO SOP #WP-01, #WP-04	SW 846 Method 8270C / S-IN-O-068-rev.12	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
***Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Soil
Analytical Group	Polychlorinated bi phenyls (PCBs)
Concentration Level	Low to medium (µg/kg to mg/kg)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SESCO SOP #WP-01, #WP-04	SW 846 Method 8082 / S-IN-O-050-rev.11	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
***Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Soil
Analytical Group	Dioxins (TCDD)
Concentration Level	Low to medium (µg/kg to mg/kg)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SESCO SOP #WP-01, #WP-04	SW 846 Method 8082 / S-IN-O-050-rev.11	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*****Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Groundwater
Analytical Group	Metals
Concentration Level	Low (µg/L)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Geoprobe® Operations Manual (UOS 2005b, Kejr Engineering 2003)	SW 846/6020 / S-IN-O-031 Mercury 7470A / S-IN-I-040-rev.13 Cr6+ 7196A / S-IN-I-063-rev.11	Precision (field)	RPD ±20%	Field Duplicate	S & A
		Accuracy/Bias (field) Contamination	No analyte > MRL	Equipment Blank	S & A
		Precision (laboratory)	PACE QC Limits	MS/MSD or Sample and Duplicate	A
		Bias (laboratory)	PACE QC Limits	Matrix Spike	A
		Bias (laboratory)	PACE QC Limits	Laboratory Control Sample (LCS)	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*****Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Groundwater
Analytical Group	Volatile Organics
Concentration Level	Low to medium (µg/L)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Geoprobe® Operations Manual (UOS 2005b, Kejr Engineering 2003)	SW 846 Method 8260/5035 / S-IN-O-029-rev.17	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
***Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Groundwater
Analytical Group	Semi-Volatile Organics
Concentration Level	Low to medium (µg/L)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Geoprobe® Operations Manual (UOS 2005b, Kejr Engineering 2003)	SW 846 Method 8270C / S-IN-O-068-rev.12	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Groundwater
Analytical Group	Polychlorinated bi phenyls (PCBs)
Concentration Level	Low to medium (µg/L)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Geoprobe® Operations Manual (UOS 2005b, Kejr Engineering 2003)	SW 846 Method 8082 / S-IN-O-050-rev.11	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Groundwater
Analytical Group	Dioxins (TCDD)
Concentration Level	Low to medium (µg/L)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Geoprobe® Operations Manual (UOS 2005b, Kejr Engineering 2003)	SW 846 Method 8082 / S-IN-O-050-rev.11	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Waste Solids
Analytical Group	Metals
Concentration Level	Low to Medium (mg/L)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
SESCO SOP #WP-01, #WP-04	SW 846 Method 6020 / S-IN-O-031 Mercury 7471A / S-IN-I-040-rev.13 TCLP 1311 / S-IN-I-062-rev.10	Precision (field)	RPD ±20%	Field Duplicate	S & A
		Accuracy/Bias (field) Contamination	No analyte > MRL	Equipment Blank	S & A
		Precision (laboratory)	PACE QC Limits	MS/MSD or Sample and Duplicate	A
		Bias (laboratory)	PACE QC Limits	Matrix Spike	A
		Bias (laboratory)	PACE QC Limits	Laboratory Control Sample (LCS)	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*****Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Waste Solids
Analytical Group	Volatile Organics
Concentration Level	Low to medium (µg/L to mg/L)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SESCO SOP #WP-01, #WP-04	SW 846 Method 8260/5035 / S-IN-O-029-rev.17	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*****Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #12 – Measurement Performance Criteria Table – Field QC Samples (Cont.)

Matrix	Waste Solids
Analytical Group	Semi-Volatile Organics
Concentration Level	Low to medium (µg/L to mg/L)

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SESCO SOP #WP-01, #WP-04	SW 846 Method 8270C / S-IN-O-068-rev.12 TCLP 1311 / S-IN-I-062-rev.10	Precision (field)	±35% RPD	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	List compound specific RPD	MS/MSD*	A
		Accuracy (laboratory)	List compound specific %R	**DMCs; MS/MSD*	A

[1] Reference number from QAPP Worksheet #21
[2] Reference number from QAPP Worksheet #23
*Optional MS/MSD –**Deuterated Monitoring Compounds (DMCs) – Reference Contract Laboratory Program (CLP) SOM01.2

QAPP Worksheet #13 – Secondary Data Criteria and Limitations Table

Identify all secondary data and information that will be used for the project and their originating sources. Specify how the secondary data will be used and the limitations on their use.

☐ Worksheet Not Applicable (State Reason)

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use
Soil analytical results and field observations	Dixon Road Site Assessment, U.S. EPA	U.S. EPA Definitive data	Using field observed drum and waste locations, additional areas will be located for screening and sampling	Observed drum location may not represent or identify other buried areas

QAPP Worksheet #14 – Summary of Project Tasks

Provide a brief overview of the listed project activities.

☐ Worksheet Not Applicable (State Reason)

Sampling Tasks: Collect grab/composite soil, water and drum samples using standard techniques as required for analysis using SW-846 methods. Exact sample media, number of samples, and parameters will be determined based on project-specific data.
Analysis Tasks: A subcontracted laboratory will analyze samples for required analyses using standard EPA-approved methods.
Quality Control Tasks: Implement SOPs for sample collection, packaging, transport, and storage prior to analysis. Quality control sample handling protocols are described on Worksheet #26.
Secondary Data: Secondary data will be collected as needed
Data Management Tasks: Records generated during sample collection and analyses document the validity and authenticity of the project data. The field and laboratory data (electronic and hard copy) generated for this study will be retained at the SESCO office with electronic copies provided to the EPA. Field logs, sample records, and chain-of-custody records will be kept for a period of 5 years.
Documentation and Records: 1. Procedures, observations, and test results will be documented for all sample collection activities, laboratory analyses, and reporting. 2. Field data will be recorded in a field notebook, and will include, but not be limited to, pertinent observations and performance and maintenance indicators. Field records will be maintained during all stages of sample collection and preparation for transport to the laboratory.
Assessment/Audit Tasks: 1. The following reports may be completed if a deviation from the field sample matrix or QAPP is encountered, or to document an audit: a. Corrective action reports documenting any problems encountered during field activities and corrective actions taken b. System and performance audit reports completed during the investigation, a summary of any changes made to documented procedures, and the rationale for the changes. 2. Contractor may prepare the data quality and usability report, which would be included in the closure report. The report would summarize the results of the data validation and data quality review and would describe any significant quality assurance

QAPP Worksheet #14 – Summary of Project Tasks (Cont.)

Data Review Tasks:

1. Laboratory will perform data reduction as described in each test method for this project and will submit sample results and QA/QC results.
2. The laboratory quality assurance officer and/or laboratory director are responsible for reviewing the laboratory data and QA/QC reports, and checking data reduction prior to submittal to contractor and OSC. The laboratory will correct any transcription or computational errors identified during this review.
3. An abbreviated data validation review will be completed by contractor, as described in Worksheet #36.
4. Validated data and all related field logs/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to Project Quality Objectives and required Action Limits. Corrective action is initiated, as necessary. Final data are placed in a database with any necessary qualifiers, and tables, charts, and graphs are generated.

QAPP Worksheet #15 – Reference Limits and Evaluation Table

Complete this worksheet for each matrix, analytical group, and concentration level you encounter on your project. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Finally, list the published and achievable detection and quantitation limits for each analyte.

☐ Worksheet Not Applicable (State Reason)

Matrix: Soil
Analytical Group: Volatile Organic Compounds
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (mg/Kg)	Project Limit Goal (mg/Kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/Kg)	QLs (mg/Kg)	MDLs (mg/Kg)	QLs (mg/Kg)
Acetone	67-64-1	100,000	100,000	0.05	0.1	0.05	0.1
Acrolein	107-02-8	0.60	0.60	0.05	0.1	0.05	0.1
Acrylonitrile	107-13-1	11	11	0.05	0.1	0.05	0.1
Benzene	71-43-2	51	51	0.001	0.005	0.001	0.005
Bromobenzene	108-86-1	680	680	0.0025	0.005	0.0025	0.005
Bromodichloromethane	75-27-4	13	13	0.0025	0.005	0.0025	0.005
Bromoform	75-25-2	2,900	2,900	0.0032	0.005	0.0032	0.005
Bromomethane	74-83-9	30	30	0.004	0.005	0.004	0.005
Bromochloromethane	74-97-5	630	630	0.0025	0.005	0.0025	0.005
2-Butanone (MEK)	78-93-3	28,000	28,000	0.012	0.025	0.012	0.025
n-Butylbenzene	104-51-8	110	110	0.0025	0.005	0.0025	0.005
sec-Butylbenzene	135-98-8	150	150	0.0025	0.005	0.0025	0.005
tert-Butylbenzene	98-06-6	180	180	0.0025	0.005	0.0025	0.005
Carbon disulfide	75-15-0	740	740	0.005	0.01	0.005	0.01
Carbon tetrachloride	56-23-5	29	29	0.0025	0.005	0.0025	0.005
Chlorobenzene	108-90-7	760	760	0.0025	0.005	0.0025	0.005
Chloroethane	NL	NL	NL	0.0025	0.005	0.0025	0.005
Chloroform	67-66-3	14	14	0.0025	0.005	0.0025	0.005

Chloromethane	74-87-3	460	460	0.0025	0.005	0.0025	0.005
2-Chlorotoluene	95-49-8	910	910	0.0025	0.005	0.0025	0.005
4-Chlorotoluene	106-43-4	250	250	0.0025	0.005	0.0025	0.005
Dibromochloromethane	124-48-1	32	32	0.0025	0.005	0.0025	0.005
1,2-Dibromoethane (EDB)	106-93-4	1.6	1.6	0.0025	0.005	0.0025	0.005
Dibromomethane	74-95-3	98	98	0.0032	0.005	0.0032	0.005
1,2-Dichlorobenzene	95-50-1	380	380	0.0025	0.005	0.0025	0.005
1,3-Dichlorobenzene	NL	NL	NL	0.0025	0.005	0.0025	0.005
1,4-Dichlorobenzene	106-46-7	110	110	0.0025	0.005	0.0025	0.005
Dichlorodifluoromethane	75-71-8	370	370	0.0025	0.005	0.0025	0.005
1,1-Dichloroethane	75-34-3	160	160	0.0025	0.005	0.0025	0.005
1,2-Dichloroethane	107-06-2	20	20	0.0025	0.005	0.0025	0.005
1,1-Dichloroethene	75-35-4	1,000	1,000	0.0025	0.005	0.0025	0.005
cis-1,2-Dichloroethene	156-59-2	2,300	2,300	0.0025	0.005	0.0025	0.005
trans-1,2-Dichloroethene	156-60-5	1,700	1,700	0.0025	0.005	0.0025	0.005
1,2-Dichloropropane	78-87-5	44	44	0.0025	0.005	0.0025	0.005
1,3-Dichloropropane	142-28-9	1,500	1,500	0.0025	0.005	0.0025	0.005
2,2-Dichloropropane	NL	NL	NL	0.0025	0.005	0.0025	0.005
1,1-Dichloropropene	NL	NL	NL	0.0025	0.005	0.0025	0.005
cis-1,3-Dichloropropene	NL	NL	NL	0.0025	0.005	0.0025	0.005
trans-1,3-Dichloropropene	NL	NL	NL	0.0025	0.005	0.0025	0.005
Ethylbenzene	100-41-4	250	250	0.0025	0.005	0.0025	0.005
Ethyl methacrylate	97-63-2	1,100	1,100	0.05	0.1	0.05	0.1
Hexachloro-1,3-butadiene	NL	NL	NL	0.0025	0.005	0.0025	0.005
2-Hexanone	591-78-6	1,300	1,300	0.005	0.1	0.005	0.1
Iodomethane	NL	NL	NL	0.005	0.1	0.005	0.1
Isopropylbenzene (Cumene)	98-82-8	270	270	0.0025	0.005	0.0025	0.005
p-Isopropyltoluene	NL	NL	NL	0.0025	0.005	0.0025	0.005
Methylene Chloride	75-09-2	3,200	3,200	0.01	0.02	0.01	0.02

4-Methyl-2-pentanone (MIBK)	NL	NL	NL	0.012	0.03	0.012	0.03
1-Methylnaphthalene	90-12-0	730	730	0.01	0.01	0.01	0.01
2-Methylnaphthalene	91-57-6	3,000	3,000	0.01	0.01	0.01	0.01
Methyl-tert-butyl-Ether (MTBE)	1634-04-4	2,100	2,100	0.0025	0.005	0.0025	0.005
Naphthalene	91-20-3	170	170	0.0025	0.005	0.0025	0.005
n-Propylbenzene	103-65-1	260	260	0.0025	0.005	0.0025	0.005
Styrene	100-42-5	870	870	0.0025	0.005	0.0025	0.005
1,1,1,2-Tetrachloroethane	630-20-6	88	88	0.0025	0.005	0.0025	0.005
1,1,2,2-Tetrachloroethane	79-34-5	27	27	0.0025	0.005	0.0025	0.005
Tetrachloroethene	127-18-4	170	170	0.0014	0.005	0.0014	0.005
Toluene	108-88-3	820	820	0.0025	0.005	0.0025	0.005
trans-1,4-Dichloro-2-butene	110-57-6	0.32	0.32	0.05	0.1	0.05	0.1
1,2,3-Trichlorobenzene	87-61-6	660	660	0.0025	0.005	0.0025	0.005
1,2,4-Trichlorobenzene	120-82-1	260	260	0.0025	0.005	0.0025	0.005
1,1,1-Trichloroethane	71-55-6	640	640	0.0025	0.005	0.0025	0.005
1,1,2-Trichloroethane	79-00-5	6.3	6.3	0.0025	0.005	0.0025	0.005
Trichloroethene	79-01-6	19	19	0.001	0.005	0.001	0.005
Trichlorofluoromethane	75-69-4	1,200	1,200	0.0025	0.005	0.0025	0.005
1,2,3-Trichloropropane	96-18-4	1.1	1.1	0.0025	0.005	0.0025	0.005
1,2,4-Trimethylbenzene	95-63-6	220	220	0.0025	0.005	0.0025	0.005
1,3,5-Trimethylbenzene	108-67-8	180	180	0.0025	0.005	0.0025	0.005
Vinyl Acetate	108-05-4	2,800	2,800	0.05	0.1	0.05	0.1
Vinyl chloride	75-01-4	17	17	0.005	0.0025	0.005	0.0025
Xylenes, Total	1330-20-7	260	260	0.005	0.01	0.005	0.01

Matrix: Soil
Analytical Group: Semi-Volatile Organic Compounds
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (mg/Kg)	Project Limit Goal (mg/Kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/Kg)	QLs (mg/Kg)	MDLs (mg/Kg)	QLs (mg/Kg)
Acenaphthene	83-32-9	45,000	45,000	0.16	0.33	0.16	0.33
Acenaphthylene	NL	NL	NL	0.16	0.33	0.16	0.33
Anthracene	120-12-7	100,000	100,000	0.16	0.33	0.16	0.33
Benzo[a]anthracene	56-55-3	29	29	0.16	0.33	0.16	0.33
Benzo[a]pyrene	50-32-8	2.9	2.9	0.16	0.33	0.16	0.33
Benzo[b]fluoranthene	205-99-2	29	29	0.16	0.33	0.16	0.33
Benzo[k]fluoranthene	2007-08-9	290	290	0.16	0.33	0.16	0.33
Benzo[g,h,i]perylene	NL	NL	NL	0.16	0.33	0.16	0.33
Benzyl alcohol	100-51-6	82,000	82,000	0.33	0.66	0.33	0.66
Bis(2-chloroethoxy) methane	111-91-1	2,500	2,500	0.16	0.33	0.16	0.33
Bis(2-chloroethyl) ether	111-44-4	10	10	0.16	0.33	0.16	0.33
Bis(2-ethylhexyl) phthalate	117-81-7	1,600	1,600	0.16	0.33	0.16	0.33
Bis(2-chloroisopropyl) ether	NL	NL	NL	0.16	0.33	0.16	0.33
4-Bromophenyl phenyl ether	NL	NL	NL	0.16	0.33	0.16	0.33
Butyl benzyl phthalate	85-68-7	12,000	12,000	0.16	0.33	0.16	0.33
Caprolactam	105-60-2	100,000	100,000	0.16	0.33	0.16	0.33
4-Chloroaniline	NL	NL	NL	0.33	0.66	0.33	0.66
4-Chloro-3-methylphenol	NL	NL	NL	0.33	0.66	0.33	0.66
2-Chloronaphthalene	NL	NL	NL	0.16	0.33	0.16	0.33
2-Chlorophenol	95-57-8	5,800	5,800	0.16	0.33	0.16	0.33
4-Chlorophenyl phenyl ether	NL	NL	NL	0.16	0.33	0.16	0.33
Chrysene	218-01-9	2,900	2,900	0.16	0.33	0.16	0.33
Dibenz[a,h]anthracene	53-70-3	2.9	2.9	0.16	0.33	0.16	0.33
Dibenzofuran	132-64-9	1,000	1,000	0.16	0.33	0.16	0.33

Di-n-butyl phthalate	NL	NL	NL	0.16	0.33	0.16	0.33
3,3'-Dichlorobenzidine	91-94-1	51	51	0.33	0.66	0.33	0.66
2,4-Dichlorophenol	120-83-2	2,500	2,500	0.16	0.33	0.16	0.33
Diethyl phthalate	84-66-2	100,000	100,000	0.16	0.33	0.16	0.33
2,4-Dimethylphenol	105-67-9	16,000	16,000	0.16	0.33	0.16	0.33
Dimethylphthalate	NL	NL	NL	0.16	0.33	0.16	0.33
4,6-Dinitro-2-methylphenol	NL	NL	NL	0.8	1.6	0.8	1.6
2,4-Dinitrophenol	51-28-5	1,600	1,600	0.8	1.6	0.8	1.6
2,4-Dinitrotoluene	121-14-2	74	74	0.16	0.33	0.16	0.33
2,6-Dinitrotoluene	606-20-2	15	15	0.16	0.33	0.16	0.33
Di-n-octyl phthalate	117-84-0	8,200	8,200	0.16	0.33	0.16	0.33
Fluoranthene	206-44-0	30,000	30,000	0.16	0.33	0.16	0.33
Fluorene	86-73-7	30,000	30,000	0.16	0.33	0.16	0.33
Hexachlorobenzene	118-74-1	14	14	0.16	0.33	0.16	0.33
Hexachloro-1,3-butadiene	NL	NL	NL	0.16	0.33	0.16	0.33
Hexachlorocyclopentadiene	77-47-4	4,900	4,900	0.16	0.33	0.16	0.33
Hexachloroethane	67-72-1	580	580	0.16	0.33	0.16	0.33
Indeno[1,2,3-cd]pyrene	193-39-5	29	29	0.16	0.33	0.16	0.33
Isophorone	78-59-1	24,000	24,000	0.16	0.33	0.16	0.33
2-Methylphenol	NL	NL	NL	0.16	0.33	0.16	0.33
3 & 4-Methylphenol	NL	NL	NL	0.33	0.66	0.33	0.66
2-Methylnaphthalene	91-57-6	3,000	3,000	0.16	0.33	0.16	0.33
N-Nitroso-di-n-propylamine	621-64-7	3.3	3.3	0.16	0.33	0.16	0.33
N-Nitrosodiphenylamine	86-30-6	4,700	4,700	0.16	0.33	0.16	0.33
Naphthalene	91-20-3	170	170	0.16	0.33	0.16	0.33
2-Nitroaniline	88-74-4	8,000	8,000	0.8	1.6	0.8	1.6
3-Nitroaniline	NL	NL	NL	0.8	1.6	0.8	1.6
4-Nitroaniline	100-01-6	1,200	1,200	0.8	1.6	0.8	1.6
Nitrobenzene	98-95-3	220	220	0.16	0.33	0.16	0.33
2-Nitrophenol	NL	NL	NL	0.16	0.33	0.16	0.33

4-Nitrophenol	NL	NL	NL	0.8	1.6	0.8	1.6
Pentachlorophenol	87-86-5	40	40	0.8	1.6	0.8	1.6
Phenanthrene	NL	NL	NL	0.16	0.33	0.16	0.33
Phenol	108-95-2	100,000	100,000	0.16	0.33	0.16	0.33
Pyrene	129-00-0	23,000	23,000	0.16	0.33	0.16	0.33
2,4,5-Trichlorophenol	95-95-4	82,000	82,000	0.16	0.33	0.16	0.33
2,4,6-Trichlorophenol	88-06-2	820	820	0.16	0.33	0.16	0.33

Matrix: Soil
Analytical Group: Metals
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (mg/Kg)	Project Limit Goal (mg/Kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/Kg)	QLs (mg/Kg)	MDLs (mg/Kg)	QLs (mg/Kg)
Lead	7439-92-1	800	800	1	2	1	2
Arsenic	7440-38-2	30	30	1	2	1	2
Chromium (III)	16065-83-1	100,000	100,000	1	2	1	2
Chromium (VI)	18540-29-9	63	63	1	2	1	2
Zinc	7440-66-6	100,000	100,000	1	2	1	2
Mercury	7439-97-6	3.1	3.1	0.001	0.33	0.001	0.33

Matrix: Soil
Analytical Group: Polychlorinated biphenyls (PCBs)
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (mg/Kg)	Project Limit Goal (mg/Kg)	Achievable Laboratory Limits		Achievable Laboratory Limits	
				MDLs (mg/Kg)	QLs (mg/Kg)	MDLs (mg/Kg)	QLs (mg/Kg)
Aroclor 1248	12672-29-6	10	10	0.05	0.1	0.05	0.1
Aroclor 1254	11097-69-1	10	10	0.05	0.1	0.05	0.1

Matrix: Soil
Analytical Group: Dioxins
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (mg/Kg)	Project Action Limit (mg/Kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/Kg)	QLs (mg/Kg)	MDLs (mg/Kg)	QLs (mg/Kg)
TCDD	1746-01-6	0.00022	0.00022	NL	NL	NL	NL

Matrix: Groundwater
Analytical Group: Volatile Organic Compounds
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (ug/L)	Project Limit Goal (ug/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/L)	QLs (ug/L)	MDLs (ug/L)	QLs (ug/L)
Acetone	67-64-1	14,000	14,000	50	100	50	100
Acrolein	107-02-8	50	50	25	50	25	50
Acrylonitrile	107-13-1	100	100	50	100	50	100
Benzene	71-43-2	5	5	1.0	5.0	1.0	5.0
Bromobenzene	108-86-1	62	62	2.5	5.0	2.5	5.0
Bromodichloromethane	75-27-4	80	80	2.5	5.0	2.5	5.0
Bromoform	75-25-2	80	80	2.5	5.0	2.5	5.0
Bromomethane	74-83-9	7.5	7.5	3.9	5.0	3.9	5.0
Bromochloromethane	74-97-5	83	83	2.5	5.0	2.5	5.0
2-Butanone (MEK)	78-93-3	5,600	5,600	12	25	12	25
n-Butylbenzene	104-51-8	1,000	1,000	2.5	5.0	2.5	5.0
sec-Butylbenzene	135-98-8	2,000	2,000	2.5	5.0	2.5	5.0
tert-Butylbenzene	98-06-6	690	690	2.5	5.0	2.5	5.0
Carbon disulfide	75-15-0	810	810	5.0	10	5.0	10
Carbon tetrachloride	56-23-5	5	5	2.5	5.0	2.5	5.0
Chlorobenzene	108-90-7	100	100	2.5	5.0	2.5	5.0
Chloroethane	NL	NL	NL	2.5	5.0	2.5	5.0
Chloroform	67-66-3	80	80	2.5	5.0	2.5	5.0
Chloromethane	74-87-3	190	190	2.5	5.0	2.5	5.0
2-Chlorotoluene	NL	NL	NL	2.5	5.0	2.5	5.0
4-Chlorotoluene	NL	NL	NL	2.5	5.0	2.5	5.0
Dibromochloromethane	124-48-1	80	80	2.5	5.0	2.5	5.0
1,2-Dibromoethane (EDB)	106-93-4	5	5	2.5	5.0	2.5	5.0
Dibromomethane	74-95-3	8	8	2.7	5.0	2.7	5.0
1,2-Dichlorobenzene	95-50-1	600	600	2.5	5.0	2.5	5.0

1,3-Dichlorobenzene	NL	NL	NL	2.5	5.0	2.5	5.0
1,4-Dichlorobenzene	106-46-7	75	75	2.5	5.0	2.5	5.0
Dichlorodifluoromethane	75-71-8	200	200	5.0	5.0	5.0	5.0
1,1-Dichloroethane	75-34-3	27	27	2.5	5.0	2.5	5.0
1,2-Dichloroethane	107-06-2	5	5	2.5	5.0	2.5	5.0
1,1-Dichloroethene	75-35-4	7	7	2.5	5.0	2.5	5.0
cis-1,2-Dichloroethene	156-59-2	70	70	2.5	5.0	2.5	5.0
trans-1,2-Dichloroethene	156-60-5	100	100	2.5	5.0	2.5	5.0
1,2-Dichloropropane	78-87-5	5	5	2.5	5.0	2.5	5.0
1,3-Dichloropropane	142-28-9	370	370	2.5	5.0	2.5	5.0
2,2-Dichloropropane	NL	NL	NL	4.2	5.0	4.2	5.0
1,1-Dichloropropene	NL	NL	NL	2.5	5.0	2.5	5.0
cis-1,3-Dichloropropene	NL	NL	NL	2.5	5.0	2.5	5.0
trans-1,3-Dichloropropene	NL	NL	NL	2.5	5.0	2.5	5.0
Ethylbenzene	100-41-4	700	700	2.5	5.0	2.5	5.0
Ethyl methacrylate	97-63-2	460	460	50	100	50	100
Hexachloro-1,3-butadiene	NL	NL	NL	2.5	5.0	2.5	5.0
2-Hexanone	591-78-6	38	38	12	25	12	25
Iodomethane	NL	NL	NL	6.8	10	6.8	10
Isopropylbenzene (Cumene)	98-82-8	450	450	2.5	5.0	2.5	5.0
p-Isopropyltoluene	NL	NL	NL	2.5	5.0	2.5	5.0
Methylene Chloride	75-09-2	5	5	3.2	5.0	3.2	5.0
4-Methyl-2-pentanone (MIBK)	108-10-1	1,200	1,200	12	25	12	25
1-Methylnaphthalene	90-12-0	11	11	5.0	5.0	5.0	5.0
2-Methylnaphthalene	91-57-6	36	36	10	10	10	10
Methyl-tert-butyl-Ether (MTBE)	1634-04-4	140	140	2.1	4.0	2.1	4.0
Napthalene	91-20-3	1.7	1.7	1.4	1.4	1.4	1.4
n-Propylbenzene	103-65-1	660	660	2.5	5.0	2.5	5.0
Styrene	100-42-5	100	100	2.5	5.0	2.5	5.0

1,1,1,2-Tetrachloroethane	630-20-6	5.7	5.7	2.5	5.0	2.5	5.0
1,1,2,2-Tetrachloroethane	79-34-5	5	5	2.5	5.0	2.5	5.0
Tetrachloroethene	127-18-4	5	5	1.2	5.0	1.2	5.0
Toluene	108-88-3	1,000	1,000	2.5	5.0	2.5	5.0
trans-1,4-Dichloro-2-butene	110-57-6	100	100	50	100	50	100
1,2,3-Trichlorobenzene	87-61-6	7	7	2.5	5.0	2.5	5.0
1,2,4-Trichlorobenzene	120-82-1	70	70	2.5	5.0	2.5	5.0
1,1,1-Trichloroethane	71-55-6	200	200	2.5	5.0	2.5	5.0
1,1,2-Trichloroethane	79-00-5	5	5	2.5	5.0	2.5	5.0
Trichloroethene	79-01-6	5	5	1.9	5.0	1.9	5.0
Trichlorofluoromethane	75-69-4	1,100	1,100	2.5	5.0	2.5	5.0
1,2,3-Trichloropropane	96-18-4	5	5	2.5	5.0	2.5	5.0
1,2,4-Trimethylbenzene	95-63-6	15	15	2.5	5.0	2.5	5.0
1,3,5-Trimethylbenzene	108-67-8	120	120	2.5	5.0	2.5	5.0
Vinyl Acetate	108-05-4	410	410	25	50	25	50
Vinyl chloride	75-01-4	2	2	2.0	2.0	2.0	2.0
Xylenes, Total	1330-20-7	10,000	10,000	5.0	10	5.0	10

Matrix: Groundwater
Analytical Group: Semi-Volatile Organic Compounds
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (ug/L)	Project Limit Goal (ug/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/L)	QLs (ug/L)	MDLs (ug/L)	QLs (ug/L)
Acenaphthene	83-32-9	530	530	5	10	5	10
Acenaphthylene	NL	NL	NL	5	10	5	10
Anthracene	120-12-7	1,800	1,800	5	10	5	10
Benzo[a]anthracene	56-55-3	0.34	0.34	0.05	0.1	0.05	0.1
Benzo[a]pyrene	50-32-8	0.2	0.2	0.05	0.1	0.05	0.1
Benzo[b]fluoranthene	205-99-2	0.34	0.34	0.05	0.1	0.05	0.1
Benzo[k]fluoranthene	2007-08-9	3.4	3.4	0.05	0.1	0.05	0.1
Benzo[g,h,i]perylene	NL	NL	NL	5	10	5	10
Benzyl alcohol	100-51-6	2,000	2,000	10	20	10	20
Bis(2-chloroethoxy) methane	111-91-1	59	59	5	10	5	10
Bis(2-chloroethyl) ether	111-44-4	10	10	5	10	5	10
Bis(2-ethylhexyl) phthalate	117-81-7	6	6	2.5	5	2.5	5
Bis(2-chloroisopropyl) ether	NL	NL	NL	34	5	34	5
4-Bromophenyl phenyl ether	NL	NL	NL	5	10	5	10
Butyl benzyl phthalate	85-68-7	160	160	5	10	5	10
Caprolactam	105-60-2	9,900	9,900	10	20	10	20
4-Chloroaniline	NL	NL	NL	10	20	10	20
4-Chloro-3-methylphenol	NL	NL	NL	10	20	10	20
2-Chloronaphthalene	NL	NL	NL	5	10	5	10
2-Chlorophenol	95-57-8	91	91	5	10	5	10
4-Chlorophenyl phenyl ether	NL	NL	NL	5	10	5	10
Chrysene	218-01-9	34	34	5	10	5	10
Dibenz[a,h]anthracene	53-70-3	0.1	0.1	0.05	0.1	0.05	0.1
Dibenzofuran	132-64-9	7.9	7.9	5	10	5	10

Di-n-butyl phthalate	NL	NL	NL	5	10	5	10
3,3'-Dichlorobenzidine	91-94-1	20	20	10	20	10	20
2,4-Dichlorophenol	120-83-2	46	46	5	10	5	10
Diethyl phthalate	84-66-2	15,000	15,000	5	10	5	10
2,4-Dimethylphenol	105-67-9	360	360	5	10	5	10
Dimethylphthalate	NL	NL	NL	5	10	5	10
4,6-Dinitro-2-methylphenol	NL	NL	NL	25	50	25	50
2,4-Dinitrophenol	51-28-5	39	39	50	50	50	50
2,4-Dinitrotoluene	121-14-2	10	10	5	10	5	10
2,6-Dinitrotoluene	606-20-2	10	10	5	10	5	10
Di-n-octyl phthalate	117-84-0	200	200	5	10	5	10
Fluoranthene	206-44-0	800	800	5	10	5	10
Fluorene	86-73-7	290	290	5	10	5	10
Hexachlorobenzene	118-74-1	10	10	5	10	5	10
Hexachloro-1,3-butadiene	NL	NL	NL	2.5	5	2.5	5
Hexachlorocyclopentadiene	77-47-4	50	50	20	20	20	20
Hexachloroethane	67-72-1	6.9	6.9	5	10	5	10
Indeno[1,2,3-cd]pyrene	193-39-5	0.34	0.34	0.05	0.1	0.05	0.1
Isophorone	78-59-1	780	780	5	10	5	10
2-Methylphenol	NL	NL	NL	5	10	5	10
3 & 4-Methylphenol	NL	NL	NL	10	20	10	20
2-Methylnaphthalene	91-57-6	36	36	5	10	5	10
N-Nitroso-di-n-propylamine	621-64-7	10	10	5	10	5	10
N-Nitrosodiphenylamine	86-30-6	120	120	5	10	5	10
Naphthalene	91-20-3	1.7	1.7	0.5	1	0.5	1
2-Nitroaniline	88-74-4	190	190	25	50	25	50
3-Nitroaniline	NL	NL	NL	25	50	25	50
4-Nitroaniline	100-01-6	38	38	25	50	25	50
Nitrobenzene	98-95-3	10	10	5	10	5	10
2-Nitrophenol	NL	NL	NL	5	10	5	10

4-Nitrophenol	NL	NL	NL	50	50	50	50
Pentachlorophenol	87-86-5	50	50	25	50	25	50
Phenanthrene	NL	NL	NL	5	10	5	10
Phenol	108-95-2	5,800	5,800	5	10	5	10
Pyrene	129-00-0	120	120	5	10	5	10
2,4,5-Trichlorophenol	95-95-4	1,200	1,200	5	10	5	10
2,4,6-Trichlorophenol	88-06-2	12	12	5	10	5	10

Matrix: Groundwater
Analytical Group: Metals
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (ug/L)	Project Limit Goal (ug/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/L)	QLs (ug/L)	MDLs (ug/L)	QLs (ug/L)
Lead	7439-92-1	15	15	2.94	10	2.94	10
Arsenic	7440-38-2	10	10	2.93	10	2.93	10
Chromium (III)	16065-83-1	22,000	22,000	2.49	10	2.49	10
Chromium (VI)	18540-29-9	0.35	0.35				
Zinc	7440-66-6	6,000	6,000	2.98	20	2.98	20
Mercury	7439-97-6	2	2	0.012	2	0.012	2

Matrix: Groundwater
Analytical Group: Polychlorinated biphenyls (PCBs)
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (ug/L)	Project Limit Goal (ug/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/L)	QLs (ug/L)	MDLs (ug/L)	QLs (ug/L)
Aroclor 1248	12672-29-6	0.39	0.39	0.036	0.1	0.036	0.1
Aroclor 1254	11097-69-1	0.39	0.39	0.035	0.1	0.035	0.1

Matrix: Groundwater
Analytical Group: Dioxins
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (ug/L)	Project Action Limit (ug/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/L)	QLs (mg/L)	MDLs (mg/L)	QLs (mg/L)
TCDD	1746-01-6	0.00003	0.00003	NL	NL	NL	NL

Matrix: Waste
Analytical Group: TCLP Volatile Organic Compounds
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (mg/L)	Project Limit Goal (mg/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/L)	QLs (mg/L)	MDLs (mg/L)	QLs (mg/L)
Benzene	NA	0.5	0.5	0.01	0.05	0.01	0.05
Carbon tetrachloride	NA	0.5	0.5	0.025	0.05	0.025	0.05
Chlorobenzene	NA	100	100	0.001	0.005	0.001	0.005
Chloroform	NA	6	6	0.0025	0.005	0.0025	0.005
1,2-Dichloroethane	NA	0.5	0.5	0.0032	0.005	0.0032	0.005
1,1-Dichloroethene	NA	0.7	0.7	0.004	0.005	0.004	0.005
Methyl ethyl ketone	NA	200	200	0.5	1	0.5	1
Tetrachloroethene	NA	0.7	0.7	0.025	0.05	0.025	0.05
Trichloroethene	NA	0.5	0.5	0.025	0.05	0.025	0.05
Vinyl chloride	NA	0.2	0.2	0.01	0.02	0.01	0.02

Matrix: Waste
Analytical Group: TCLP Semi-Volatile Organic Compounds
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (mg/L)	Project Limit Goal (mg/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/L)	QLs (mg/L)	MDLs (mg/L)	QLs (mg/L)
2-Methylphenol (o-Cresol)	NA	200	200	0.05	0.1	0.05	0.1
3&4-Methylphenol (m&p-Cresol)	NA	200	200	0.2	0.1	0.2	0.1
1,4-Dichlorobenzene	NA	7.5	7.5	0.05	0.1	0.05	0.1
2,4-Dinitrotoluene	NA	0.13	0.13	0.05	0.1	0.05	0.1
Hexachlorobenzene	NA	0.13	0.13	0.05	0.1	0.05	0.1
Hexachlorobutadiene	NA	0.5	0.5	0.05	0.1	0.05	0.1
Hexachloroethane	NA	3	3	0.05	0.1	0.05	0.1
Nitrobenzene	NA	2	2	0.05	0.1	0.05	0.1
Pentachlorophenol	NA	100	100	0.25	0.5	0.25	0.5

Pyridine	NA	5	5	0.05	0.1	0.05	0.1
2,4,5-Trichlorophenol	NA	400	400	0.25	0.5	0.25	0.5
2,4,6-Trichlorophenol	NA	2	2	0.05	0.1	0.05	0.1

Matrix: Waste
Analytical Group: TCLP Metals
Concentration Level: Low to medium

Analyte	CAS Number	Project Action Limit (mg/L)	Project Limit Goal (mg/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/L)	QLs (mg/L)	MDLs (mg/L)	QLs (mg/L)
Arsenic	NA	5	5	0.05	0.1	0.05	0.1
Barium	NA	100	100	2.5	5	2.5	5
Cadmium	NA	1	1	0.1	0.05	0.1	0.05
Chromium	NA	5	5	0.05	0.1	0.05	0.1
Lead	NA	5	5	0.05	0.1	0.05	0.1
Mercury	NA	0.2	0.2	0.003	0.006	0.003	0.006
Selenium	NA	1	1	0.05	0.1	0.05	0.1
Silver	NA	5	5	0.25	0.5	0.25	0.5

QAPP Worksheet #16 – Project Schedule / Timeline Table

List all project activities as well as the QA assessments that will be performed during the course of the project. Include the anticipated start and completion dates.

☐ Worksheet Not Applicable (State Reason)

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Field Activities	SESCO	TBD	21 Days Actual dates TBD	None	N/A
Analytical Activities	SESCO	TBD	21 Days from sample submission	Analytical Results Report	3 weeks from the receipt of the data
Drum(s) Disposal	SESCO	TBD- Initiate on the day of the receipt of analytical results	2 Weeks from the receipt of analytical results	Waste Disposal Summary Table	1 Week after disposal
Report	SESCO	3 weeks from the receipt of the data	6 weeks from the receipt of the data	Assessment Report	3 months from field activities initiation

See following page for estimated schedule of activities

Estimated Schedule

Dixon Road Site
1114 South Dixon Road
Kokomo, Indiana 46901
Site Spill Identification Number: C5M5
Administrative Settlement Agreement and Order on Consent for Removal Action Docket Number V-W-15•C-021

Order #	Milestone	Start Date	Duration (days)	End Date
89	Effective Date (ED)	4/7/2015		
16b	Site Security			
12	Contractor Notification, including Quality Management Plan		ED + 5 BD	4/14/2015
13	Project Coordinator Notification		ED + 5 BD	4/14/2015
18	HASP		ED + 30 CD	5/7/2015
17a	Work Plan, Including QAPP		ED + 30 CD	5/7/2015
17a	USEPA Review of Work Plan, HASP, & QAPP		30 CD	6/6/2015
17b	Work Plan Revisions	6/6/2015	17	6/23/2015
16c	**Field Investigation:**			
	Site Boundary Survey	7/23/2015	14	8/6/2015
	Geophysical Survey & Data Analysis	7/27/2015	21	8/17/2015
	Utility Clearance	8/3/2015	1	8/3/2015
	Surface and Sub-Surface Soil Sampling & Groundwater Sampling	8/4/2015	15	8/19/2015
	Laboratory Analysis of Soil and Groundwater Samples	8/19/2015	21	9/2/2015
	Data Validation	9/9/2015	35	10/14/2015
	Geophysical Survey Report due to EPA	8/31/2015	1	8/31/2015
	EPA review of geophysical report & test pit recommendations	8/17/2015	14	8/31/2015
	Laboratory Analytical Tables & Lab Data due to EPA	10/2/2015	1	10/2/2015
	Test Pit Excavations	9/21/2015	4	9/24/2015
	Laboratory Analysis of Soil Samples - Test Pits	9/24/2015	21	10/15/2015
	Data Validation	10/22/2015	35	11/26/2015
	Investigation Report	10/15/2015	60	12/14/2015
22	Final Report, to be drafted following all completed work	TBD	-	TBD
16e	Sampling Notification to EPA, 5 days prior		5	
21a	Progress Reports - 30th of every month			

BD = Business Days CD = Calendar Days TBD = To Be Determined

QAPP Worksheet #17 – Sampling Design and Rationale

Describe the project sampling approach. Provide the rationale for selecting sample locations and matrices for each analytical group and concentration level.

☐ Worksheet Not Applicable (State Reason)

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):

The strategy for sampling at the Dixon Road Site is to use a random-start equilateral triangular gridded sampling approach to determine the lateral extent, and judgmental stratified sampling using a Geoprobe® or test pits to determine the vertical extent of contamination. Up to two subsurface samples will be collected from each boring location: one at the bottom of the boring, and the other(s) at intervals of elevated instrument readings and/or visual and olfactory judgment.

A random-start equilateral triangular gridded system will be created to collect 45 surface soil samples across the Site. Data will be used to evaluate direct public exposure risk.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]:

See Worksheet # 18 for details.

QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table

List all site locations that will be sampled and include sample/ID number, if available. (Provide a range of sampling locations or ID numbers if a site has a large number.) Specify matrix and, if applicable, depth at which samples will be taken. Only a short reference for the sampling location rationale is necessary for the table. The text of the QAPP should clearly identify the detailed rationale associated with each reference. Complete all required information, using additional worksheets if necessary.

☐ Worksheet Not Applicable (State Reason)

Sampling Location/ID Number	Matrix	Depth (bgs)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
DR-SS010 Thru DR-SS054	Soil	0-6 inches	Metals, SVOCs, PCBs, Dioxin	Low to Medium	44+5	S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-040, S-IN-I-019, S-IN-I-031, S-IN-O-029, S-IN-O-054, S-IN-O-068, S-IN-O-163, S-IN-O-133, S-IN-O-161, S-IN-O-050, S-IN-I-063-rev.11	Grid based sampling to identify contamination
DR-WD001	Solid	Compo site	Metals, VOCs, SVOCs, PCBs, Dioxin	Medium to High	TBD	S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-040, S-IN-I-019, S-IN-I-031, S-IN-O-029, S-IN-O-054, S-IN-O-068, S-IN-O-163, S-IN-O-133, S-IN-O-161, S-IN-O-050, S-IN-I-063-rev.11	Sampling for disposal characteristics
KD-SB010 Thru KD-SB030	Soil	To ground-water	Metals, VOCs, SVOCs, PCBs, Dioxin	Medium	90+9	S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-040, S-IN-I-019, S-IN-I-031, S-IN-O-029, S-IN-O-054, S-IN-O-068, S-IN-O-163, S-IN-O-133, S-IN-O-161, S-IN-O-050, S-IN-I-063-rev.11	Random-start equilateral triangular gridded sampling approach to define the extent of contamination.
KD-GW001- thru TBD	Groundwater	NA	Metals, VOCs, SVOCs, PCBs, Dioxin	Low	TBD	S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-040, S-IN-I-019, S-IN-I-031, S-IN-O-029, S-IN-O-054, S-IN-O-068, S-IN-O-163, S-IN-O-133, S-IN-O-161, S-IN-O-050, S-IN-I-063-rev.11	Co-located with subsurface soil.

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet_#21).

QAPP Worksheet #19 – Analytical SOP Requirements Table

For each matrix, analytical group, and concentration level, list the analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time.

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Soil / Solid	Volatile Organics	Low/Medium	8260-5030/5035 S-IN-O-029, S-IN-O-054	40-ml	Terra Core, 3 40 mL vials	Terra Core Methanol: Method-40-ml Glass vials, PTFE septa cap; Low Level Method: 40-ml Glass vials, PTFE septa cap & stir bar	Terra Core: 48 hours to preserve, 14 days to analysis; Methanol Method: 14 days; Low Level Method: 14 days
Soil / Solid	Semivolatile Organics	Low/Medium	8270-3510/352/3540/ 3541/3550 S-IN-O-068, S-IN-O-163, S-IN-O-133	8-oz	1 8-oz Clear Wide Mouth Glass bottle	Cool 4oC	14 days to extract, 40 days to analysis
Soil / Solid	Dioxins & Furans 2,3,7,8 – TCDD	Low/Medium	8280/8290 S-IN-O-161, S-IN-O-050	8-oz	1 8-oz Clear Wide Mouth Glass bottle	Cool 4oC	30 days to extract, 45 days to analysis
Soil / Solid	PCBs / Pesticides	Low/Medium	8082/8081 S-IN-O-161, S-IN-O-050	8-oz	1 8-oz Clear Wide Mouth Glass bottle	Cool 4oC	14 days to extract, 40 days to analysis

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Soil / Solid	Metals (Except Mercury)	Low/Medium	6010/6020-3005/3010/3015/3050/3051 S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-019, S-IN-I-031	8-oz	1 8-oz Clear Wide Mouth Glass bottle	Cool 4oC	6 months
Soil / Solid	Mercury	Low/Medium	7470/7471 S-IN-I-040	8-oz	1 8-oz Clear Wide Mouth Glass bottle	Cool 4oC	28 days
Soil / Solid	Hex. Chromium	Low/Medium	218.6/7196A S-IN-I-063-rev.11	4-oz	1 4-oz Glass bottle	Cool to 4oC	30 days to digest, 7 days to analyzed digestates
Soil / Solid	TCLP Semivolatile Fraction	Low/Medium	1311/8270-1310 S-IN-O-068, S-IN-O-163, S-IN-O-133, S-IN-I-062	16-oz	1 16-oz Clear Wide Mouth Glass bottle	Cool 4oC	14 days to TCLP, 7 days to extract, 40 days to analysis
Soil / Solid	TCLP volatile Fraction	Low/Medium	1311/8260-1310 S-IN-O-029, S-IN-O-054, S-IN-I-062	4-oz	1 4-oz Glass PTFE lined septa bottle	Cool 4oC	14 days to TCLP, 14 days to analysis
Soil / Solid	TCLP Inorganic Fraction (all other metals)	Low/medium	1311/6010-1310 S-IN-O-130, S-IN-I-030, S-IN-I-019, S-IN-I-031, S-IN-I-062	4-oz	1 4-oz Clear Wide Mouth Glass bottle	Cool 4oC	180 days to TCLP, 180 days to analysis

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Soil / Solid	TCLP Inorganic Fraction (Hg)	Low/medium	1311/7470-1310 S-IN-I-040, S-IN-I-062	4-oz	1 4-oz Clear Wide Mouth Glass bottle	Cool 4oC	28 days to TCLP, 28 days to analysis
Soil	Hydrogen Ion (pH)	Low/medium	9040/9041	8-oz	1 8-oz Clear Wide Mouth Glass bottle	Cool 4oC	Requires analysis right away
Soil / Solid	Corrosivity	Low/medium	1110	4-oz	1 4-oz Clear Wide Mouth Glass bottle	None	14 Days
Soil / Solid	Flashpoint ASTM D9379	Low/medium	1010/1030	4-oz	1 4-oz Clear Wide Mouth Glass bottle	None	14 Days
Soil / Solid	Total Organic Halogens (TOX)	Low/medium	9020	4-oz	1 4-oz Clear Wide Mouth Glass bottle	Cool 4oC	28 days

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet_#23).

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Waste / Liquid	Volatile Organics	Low/Medium	8260-5030/5035 S-IN-O-029, S-IN-O-054	40-ml X 2	2 40-ml Glass vials, PTFE septa cap	Cool 4°C, (.008% $Na_2S_2O_3$ if residual Cl_2 present). No headspace, HCl to pH < 2	14 days
Waste / Liquid	Semivolatile Organics	Low/Medium	8270-3510/352/3540/3541/ 3550 S-IN-O-068, S-IN-O-163, S-IN-O-133	2 Liters	2 1-Liter Amber Glass bottle	Cool 4°C, (.008% $Na_2S_2O_3$ if residual Cl_2 present	7 days to extract, 40 days to analysis
Waste / Liquid	Dioxins & Furans 2,3,7,8 – TCDD	Low/Medium	8280/8290 S-IN-O-161, S-IN-O-050	2 Liters	2 1-Liter Amber Glass bottle	Cool 4°C, (.008% $Na_2S_2O_3$ if residual Cl_2 present	30 days to extract, 45 days to analysis
Waste / Liquid	PCBs / Pesticides	Low/Medium	8082/8081 S-IN-O-161, S-IN-O-050	2 Liters	2 1-Liter Amber Glass bottle	Cool 4oC	7 days to extract, 40 days to analysis
Waste / Liquid	Metals (Except Mercury and Hexavalent Chromium)	Low/Medium	6010/6020-3005/3010/3015/3050/3051 S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-019, S-IN-I-031	1-Liter	1 1-Liter HDPE bottle	Cool 4°C, HNO_3 to pH < 2	6 months
Waste / Liquid	Mercury	Low/Medium	7470/7471 S-IN-I-040	250-ml	1 250-ml HDPE bottle	Cool 4°C, HNO_3 to pH < 2	28 days

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Waste / Liquid	Hexavalent Chromium	Low/Medium	218.6 S-IN-I-063-rev.11	250-ml	1 250-ml HDPE bottle	Cool 4°C; maintain pH between 9-9.5 with buffer solution (generally NaOH or ammonium sulfate)	24 hours unless preserved per method, then 28 days
Waste / Liquid	TCLP Semivolatile Fraction	Low/Medium	1311/8270-1310 S-IN-O-068, S-IN-O-163, S-IN-O-133, S-IN-I-062	3 Liters	3 1-Liter Amber Glass bottles	Cool 4°C	7 days to extract, 40 days to analysis
Waste / Liquid	TCLP volatile Fraction	Low/Medium	1311/8260-1310 S-IN-O-029, S-IN-O-054, S-IN-I-062	500-ml	1 500-ml Glass PTFE3 lined septa bottle	Cool 4°C no headspace	14 days to analysis
Waste / Liquid	TCLP Inorganic Fraction (all other metals)	Low/medium	1311/6010-1310 S-IN-O-130, S-IN-I-030, S-IN-I-019, S-IN-I-031, S-IN-I-062	1 Liter	1 1-Liter HDPE bottle	Cool 4°C HNO3 to pH < 2	180 days to analysis
Waste / Liquid	TCLP Inorganic Fraction (Hg)	Low/medium	1311/7470-1310 S-IN-I-040, S-IN-I-062	1 Liter	1 1-Liter HDPE bottle	Cool 4°C HNO3 to pH < 2	28 days to analysis
Waste / Liquid	Corrosivity	Low/medium	1110	250 ml	1 250-ml Clear Wide Mouth Glass bottle	None	14 Days
Waste / Liquid	Flashpoint ASTM D9379	Low/medium	1010/1030	4-oz	1 4-oz Clear Wide Mouth Glass bottle	None	14 Days

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Waste / Liquid	Total Organic Halogens (TOX)	Low/medium	9020	16-oz	1 16-oz Boston Round bottle	Cool 4oC	28 days
Waste / Liquid	Hydrogen Ion (pH)	Low/medium	9040/9041	60 ml	1 60-ml HDPE	None Required	Requires analysis right away
Waste / Liquid	Paint Filter Test	Low/medium	9095B	16 oz	1 16-oz Boston Round bottle	None Required	28 days

Note

High concentration samples require cooling to Cool 4^{o}C only.
PTFE (polytetrafluoroethylene); HDPE (high-density polyethylene bottles)
Semivolatiles, PCBs/pesticides may be collected in the same container. Metals, TRPH, and cyanide may be collected in the same container. Link to all SW-846 Methods: www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm
* Collected only if VOC or SVOC analytes found during assessment activities

QAPP Worksheet #20 – Field Quality Control Sample Summary Table

Summarize by matrix, analytical group, and concentration level the number of field QC samples that will be collected and sent to the laboratory.

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Duplicate Pairs	No. of MS/MSD pairs	No. of Field Blanks	No. of Equip. Blanks	No. of Trip Blank Samples	Total No. of Samples to Lab
All Matrices	All analytical Groups	Low to High	S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-040, S-IN-I-019, S-IN-I-031, S-IN-O-029, S-IN-O-054, S-IN-O-068, S-IN-O-163, S-IN-O-133, S-IN-O-161, S-IN-O-050	To be declared in Project-Specific QAPP	One per 10 Samples	One per 20 Samples		One per week or 10 samples, whichever is more frequent	TBD	TBD
Soil / Solid	Metals	Low to Medium	S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-040, S-IN-I-019, S-IN-I-031, S-IN-I-063-rev.11	134	14	8		TBD		156
Soil / Solid	VOCs	Low to Medium	S-IN-O-029, S-IN-O-054, S-IN-I-062	135	14	8		TBD	10	167
Soil / Solid	SVOCs	Low to Medium	S-IN-O-054, S-IN-O-068, S-IN-I-062, S-IN-O-163, S-IN-O-133	135	14	8		TBD		157
Soil / Solid	PCBs	Low to Medium	S-IN-O-161, S-IN-O-050	134	14	8		TBD		156
Soil / Solid	Dioxin	Low to Medium	S-IN-O-161, S-IN-O-050	135	14	8		TBD		157

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Duplicate Pairs	No. of MS/MSD pairs	No. of Field Blanks	No. of Equip. Blanks	No. of Trip Blank Samples	Total No. of Samples to Lab
Ground Water / Decon. Water	Metals, VOCs, SVOCs, PCBs	Low	S-IN-O-130, S-IN-I-062, S-IN-I-030, S-IN-I-040, S-IN-I-019, S-IN-I-031, S-IN-O-029, S-IN-O-054, S-IN-O-068, S-IN-O-163, S-IN-O-133, S-IN-O-161, S-IN-O-050, S-IN-I-063-rev.11	1-45	1-5	1-3	One per day (as needed)	5 (one per 10 samples)	10 (1 per day of sampling)	TBD

QAPP Worksheet #21 – Project Sampling SOP References Table

List all SOPs associated with project sampling including, but not limited to, sample collection, sample preservation, equipment cleaning and decontamination, equipment testing, inspection and maintenance, supply inspection and acceptance, and sample handling and custody. Include copies of the SOPs as attachments or reference all in the QAPP. Sequentially number sampling SOP references in the Reference Number column. The reference number can be used throughout the QAPP to refer to a specific SOP.

☐ Worksheet Not Applicable (State Reason)

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
WP-02 & WP-04	Surface and sub-surface soil sampling	SESCO	Sampling tools, latex glove, etc	N	
QP-01	Drum and container management	SESCO	Drum grappler, etc	N	
WP-05	Test pit excavation	SESCO	Excavator	N	
QP-02	Field instrument calibration (PID)	SESCO	PID	N	
User's Manual	PID (Multi Rae) Operation	SESCO	Multi-RAE	N	
WP-01	Sample Chain-Of-Custody	SESCO	NA	N	
WP-03	Sample Custody Seal SOP	SESCO	NA	N	
QP-03	Field Documentation	SESCO	NA	N	
User's manual	XRF Operation/Maintenance	SESCO	XRF	N	

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

Identify all field equipment and instruments (other than analytical instrumentation) that require calibration, maintenance, testing, or inspection and provide the SOP reference number for each type of equipment. In addition, document the frequency of activity, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Frequency	Acceptance Criteria		Corrective Action	SOP	Responsible Person
Multimeter	Calibrate with standard solutions	Check battery/ expiration of Soln	NA	Daily prior to activities; if anomaly suspected	pH Meter	±0.1 unit	Clean probe, replace battery, replace membrane, replace probe	User Manual	Field Team Lead
					Dissolved Oxygen	± 3%			
					Specific conductivity	±1%			
					Temperature	±0.1 ^{0}C			
					Turbidity	±NTU			
					Others	± 10% Soln			
MultiRAE	Calibrate with Zero Air; span calibrate w/ multi-gas	Check/ replace battery/ Clean tip or bulb if necessary	Bump Test	Daily prior to activities; if anomaly suspected	LEL 52% - 64% (5%) O_2 13% - 17% (5%) H_2S 9 – 12 ppm (1ppm) CH_4 – N/A		Replace battery or Replace Unit	User Manual	Field Team Lead
MiniRae (PID)	Authorized user: Calibrate w/ zero air, then 100 ppm isobutylene (span gas)	Check battery/clean side pack and probe filter cups/ bulb if necessary	Bump Test	Daily prior to activities; if anomaly suspected	(Isobutylene): 0-2000 ±2ppm >2000ppm, ±20%		Check batteries, bulb, and filters. Service if needed	User Manual QP-02	Field Team Lead
XRF	Manuf. Required as checks indicate	Check battery, window protector	Use standardization clip	Prior to and end of day's activities; if anomaly suspected	Pass/Fail		Replace battery, re-calibrate; service	User Manual	Field Operator
Water Level Indicator	NA	Check batteries	Test button with red light indicator	Prior to day's activities	Pass/Fail		Replace batteries	NA	Field Operator

HazCat Kit	NA	Refill; expiration dates	NA	NA	NA	Replace supplies	NA	Field Team Lead

QAPP Worksheet #23 – Analytical SOP References Table

List all SOPs that will be used to perform on-site or off-site analysis. Indicate whether the procedure produces screening or definitive data. Sequentially number analytical SOP reference in the Reference Number column. Include copies of the SOPs as attachments or reference in the QAPP. The reference number can be used throughout the QAPP to refer to a specific SOP.

☐ Worksheet Not Applicable (State Reason)

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
S-IN-O-130	Microwave	NA	XRF Metal Samples	Microwave	SESCO/ER	N
S-IN-O-029	VOA 8260	Definitive	VOCs	GC/MS	Pace Analytical	N
S-IN-O-054	Separator Funnel Extraction	Definitive	VOCs/ SVOCs	Extractor	Pace Analytical	N
S-IN-O-161	Extraction of PCB Oils and Wipes	Definitive	PCBs, Oil	Extractor	Pace Analytical	N
S-IN-O-050	PCBs 8082_608	Definitive	PCBs	GC/MS	Pace Analytical	N
S-IN-O-068	BNA 8270	Definitive	BNAs	GC/MS	Pace Analytical	N
S-IN-I-062	TCLP	Definitive	Metals, VOCs, SVOCs	Extractor	Pace Analytical	N
S-IN-I -030	Metals Digestion Aqueous	Definitive	Metals	ICP	Pace Analytical	N
S-IN-I -040	Mercury 7470	Definitive	Mercury/metal	AA	Pace Analytical	N
S-IN-I -019	ICP Metals 6010	Definitive	Metals	ICP	Pace Analytical	N
S-IN-I-063-rev. 11	Hex. Chromium 7196A	Definitive	Hex. Chromium/Metals	Spectrophotometer	Pace Analytical	N

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
S-IN-I -031	Metals Digestion Solid	Definitive	Metals	ICP	Pace Analytical	N
S-IN-O-163	BNA_ PAH-SIM Combo 8270	Definitive	SVOCs	GC/MS	Pace Analytical	N
S-IN-O-133	PAH-SIM 8270	Definitive	SVOCs	GC/MS	Pace Analytical	N

QAPP Worksheet #24 – Analytical Instrument Calibration Table

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
ICP	Refer to Pace SOP	Refer to Pace SOP	Refer to Pace SOP	Refer to Pace SOP	Karl Anderson, Pace	S-IN-I-030, S-IN-I-019, S-IN-I-031
Spectrophotometer	Refer to Pace SOP	Refer to Pace SOP	Refer to Pace SOP	Refer to Pace SOP	Karl Anderson, Pace	S-IN-I-040, S-IN-I-063-rev.11
GC/MS	Refer to Pace SOP	Refer to Pace SOP	Refer to Pace SOP	Refer to Pace SOP	Karl Anderson, Pace	S-IN-O-029, S-IN-O-050, S-IN-O-163, S-IN-O-133

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

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

Identify all analytical instruments that require maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
ICP	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	For acceptable & recalibration criteria, refer to the SOP	Inspect the system, correct problem, recalibrate and/or reanalyze samples	Karl Anderson, PACE	S-IN-I-030, S-IN-I-019, S-IN-I-031
Spectrophotometer	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	For acceptable & recalibration criteria, refer to the SOP	Inspect the system, correct problem, recalibrate and/or reanalyze samples	Karl Anderson, PACE	S-IN-I-040, S-IN-I-063-rev.11
GC/MS	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	As per instrument manufacturer's requirements	For acceptable & recalibration criteria, refer to the SOP	Inspect the system, correct problem, recalibrate and/or reanalyze samples	Karl Anderson, PACE	S-IN-O-029, S-IN-O-050, S-IN-O-163, S-IN-O-133

[1]Specify the appropriate reference letter or number from Analytical SOP References table (Worksheet_#23).

QAPP Worksheet #26 – Sample Handling System

Use this worksheet to identify components of the project-specific sample handling system. Record personnel, and their organizational affiliations, who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal. Indicate the number of days field samples and their extracts/digestates will be archived prior to disposal.

☐ Worksheet Not Applicable (State Reason)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): SESCO
Sample Packaging (Personnel/Organization): SESCO
Coordination of Shipment (Personnel/Organization): SESCO
Type of Shipment/Carrier: Laboratory Courier or FedEx/UPS
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Pace Analytical Laboratory
Sample Custody and Storage (Personnel/Organization): Pace Analytical Laboratory
Sample Preparation (Personnel/Organization): Pace Analytical Laboratory
Sample Determinative Analysis (Personnel/Organization): Pace Analytical Laboratory
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): See Worksheet 17
Sample Extract/Digestate Storage (No. of days from extraction/digestion): See Worksheet 17
Biological Sample Storage (No. of days from sample collection): See Worksheet 17
SAMPLE DISPOSAL
Personnel/Organization: Pace Analytical Laboratory

QAPP Worksheet #27 - Sample Custody Requirements Table

Describe the procedures that will be used to maintain sample custody and integrity. Include examples of chain-of-custody forms, traffic reports, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms. Attach or reference applicable SOPs.

☐ Worksheet Not Applicable (State Reason)

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): The field sampler will be personally responsible for the care and custody of the samples until the samples are transferred or properly dispatched. As few people as possible will handle the samples. All sample containers will be tagged or labeled with sample identification numbers and locations, including time, and date of sample collection. Sample tags or labels for each sample can be generated by using a permanent, waterproof ink either prior to or immediately after sample collection. If freezing weather, it might be necessary to use pencil. Samples will be collected in accordance with the sampling procedures documented in the QAPP. The equipment used to collect samples will be noted, along with time of sampling, sample description, depth of sample collection, volume, and the number of sample containers. The corresponding sample identification number/label/tag will be prominently listed.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): Laboratory custody procedures for sample receiving and log in; sample storage, tracking during sample preparation and analysis; and storage of analytical data are described below: Samples submitted to the Laboratory will be accompanied by a chain-of-custody form. The chain-of-custody forms will be completed and sealed within the sample transport container, which will be opened and examined by the Laboratory Sample Custodian. The Laboratory Sample Custodian will ensure that all entries on the chain-of-custody form correspond with the sample label. If discrepancies are noted by the Laboratory Sample Custodian, project staff will be immediately contacted.

Sample Identification Procedures:

All sample containers will be tagged or labeled with sample identification numbers and locations, including time, and date of sample collection. Sample tags or labels for each sample can be generated by using a permanent, waterproof ink either prior to or immediately after sample collection. If freezing weather, it might be necessary to use pencil.

Sample numbers will be assigned in sequence and in chronological order of sample collection.

Identifier	Detail	Feature	First incremental
DRS	Dixon Road	Site	
GW SS SB DRUM	Groundwater Surface Soil Subsurface soil Drum	Matrix	GW001 SS002 SB010 DR003
###	Incremental Number	Unique Identifier	
_MMDDYY	Date		

Chain-of-custody Procedures: It is U.S. EPA Region 5 Policy to follow the U.S. EPA sample custody, or chain-or-custody protocols as described in "NEIC Policies and Procedures", EPA-330/9-78DDI-r, Revised June 1985. This custody is in three parts: Sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if they:

- are in your possession
- are in your view, after being in your possession
- are in your possession and you place them in a secured location or are in a designated secure area.

At the request of an FOSC or other designated official, PACE Chain-of-Custody form and procedures will be used. The chain-of-custody (COC) form is used to track and document unbroken custody of samples as identified by the unique sample number. The PACE standard form is shown in Exhibit 8-3. Blank forms can be obtained by contacting laboratory staff personnel. The original COC form will be kept by the receiving laboratory and will accompany the analytical report. A copy of the COC form from each group of samples will be supplied to SESCO and a copy will be placed in the project files.

Information required in filling out the Chain-of-Custody Form.

Specific project name

Project number that corresponds to the project name

Project Manager

Supervisor of sample team during sampling efforts

(SOURCE: QAPP 2001 +In order to avoid compromising sample integrity, samples for various analytes should be obtained in the following order: volatiles and total organic halide (TOX) should be taken prior to extractables; extractables should be taken prior to total metals; total metals should be taken prior to dissolved metals; dissolved metals should be taken prior to the other inorganics; other inorganics should be taken prior to radionuclides; and field measurements should be taken last. All samples are collected first from the suspected or known least contaminated area and then continuing onto the most-contaminated area).

An example of the chain-of-custody form is included in Exhibit 8-3.

QAPP Worksheet #28-A – Laboratory QC Samples Table for VOCs

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

Matrix	**Liquid/Water**
Analytical Group	VOCs
Concentration Level	Low to Medium
Sampling SOP	SESCO SOPs
Analytical Method/ SOP Reference	Refer to Work Sheet #19
Sampler's Name	TBD from SESCO and ER staff
Field Sampling Organization	SESCO/ER
Analytical Organization	Pace Analytical
No. of Sample Locations	Refer to Work Sheet #18

Lab QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**		**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**	
Method Blank	1 every 12 hours	No analyte > RQL*		Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy	No analyte > CRQL*	
Matrix Spike (Not Required)	1 per ≤ 20 samples; if requested	1,1-Dichloroethene	61-145 %R	Flag outliers	Karl Anderson, PACE	Accuracy	1,1-Dichloroethene	61-145 %R
		Benzene	76-127 %R				Benzene	76-127 %R
		Trichloroethene	71-120 %R				Trichloroethene	71-120 %R
		Toluene	76-125 %R				Toluene	76-125 %R
		Chlorobenzene	75-130 %R				Chlorobenzene	75-130 %R
Matrix Spike Duplicate (Not Required)	1 per ≤ 20 samples; if requested	1,1-Dichloroethene	0-14 %RPD	Flag outliers	Karl Anderson, PACE	Precision	1,1-Dichloroethene	0-14%RPD
		Benzene	0-11 %RPD				Benzene	0-11%RPD
		Trichloroethene	0-14 %RPD				Trichloroethene	0-14%RPD
		Toluene	0-13 %RPD				Toluene	0-13%RPD
		Chlorobenzene	0-13 %RPD				Chlorobenzene	0-13%RPD

Lab QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits		Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Deuterated Monitoring Compounds	all samples	Vinyl chloride-d3	65-131%R	Check calculations and instruments, reanalyze affected samples; *up to 3 DMCs per sample may fail to meet recovery limits	Karl Anderson, PACE	Accuracy	Vinyl chloride-d3	65-131%R
		Chloroethane-d5	71-131%R				Chloroethane-d5	71-131%R
		1,1-Dichloroethene-d2	55-104%R				1,1-Dichloroethene-d2	55-104%R
		2-Butanone-d5	49-155%R				2-Butanone-d5	49-155%R
		Chloroform-d	78-121%R				Chloroform-d	78-121%R
		1,2-Dichloroethane-d4	78-129%R				1,2-Dichloroethane-d4	78-129%R
		Benzene-d6	77-124%R				Benzene-d6	77-124%R
		1,2-Dichloropropane-d6	79-124%R				1,2-Dichloropropane-d6	79-124%R
		Toluene-d8	77-121%R				Toluene-d8	77-121%R
		trans-1,3-Dichloropropene-d4	73-121%R				trans-1,3-Dichloropropene-d4	73-121%R
		2-Hexanone-d5	28-135%R				2-Hexanone-d5	28-135%R
		1,4-Dioxane-d8	50-150%R				1,4-Dioxane-d8	50-150%R
		1,1,2,2-Tetrachloroethane-d2	73-125%R				1,1,2,2-Tetrachloroethane-d2	73-125%R
		1,2-Dichlorobenzene-d4	80-131%R				1,2-Dichlorobenzene-d4	80-131%R
Internal Standards	all samples	60-140%		Check calculations and instruments, reanalyze affected samples	Karl Anderson, PACE	Accuracy	± 40 % of response area, ± 20 sec retention time shift	

QAPP Worksheet #28-B – Laboratory QC Samples Table for VOCs

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

Matrix	**Soil/Solid**
Analytical Group	VOCs
Concentration Level	Low to Medium
Sampling SOP	SESCO SOPs
Analytical Method/ SOP Reference	Refer to Work Sheet #19
Sampler's Name	TBD from SESCO and ER staff
Field Sampling Organization	SESCO/ER
Analytical Organization	Pace Analytical
No. of Sample Locations	Refer to Work Sheet #18

Lab QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits		Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria	
Method Blank	1 every 12 hours	No analyte > CRQL*		Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy	No analyte > CRQL*	
Matrix Spike (Not Required)	1 per ≤ 20 samples; if requested	1,1-Dichloroethene	61-145 %R	Flag outliers	Karl Anderson, PACE	Accuracy	1,1-Dichloroethene	61-145 %R
		Benzene	76-127 %R				Benzene	76-127 %R
		Trichloroethene	71-120 %R				Trichloroethene	71-120 %R
		Toluene	76-125 %R				Toluene	76-125 %R
		Chlorobenzene	75-130 %R				Chlorobenzene	75-130 %R
Matrix Spike Duplicate (Not Required)	1 per < 20 samples; if requested	1,1-Dichloroethene	0-14 %RPD	Flag outliers	Karl Anderson, PACE	Precision	1,1-Dichloroethene	0-14 %RPD
		Benzene	0-11 %RPD				Benzene	0-11 %RPD
		Trichloroethene	0-14 %RPD				Trichloroethene	0-14 %RPD
		Toluene	0-13 %RPD				Toluene	0-13 %RPD
		Chlorobenzene	0-13 %RPD				Chlorobenzene	0-13 %RPD
Deuterated Monitoring Compounds	all samples	Vinyl chloride-d3	65-131 %R	Check calculations and instruments, reanalyze affected samples; *up to 3 DMCs per sample may fail to meet recovery limits	Karl Anderson, PACE	Accuracy	Vinyl chloride-d3	65-131 %R
		Chloroethane-d5	71-131 %R				Chloroethane-d5	71-131 %R
		1,1-Dichloroethene-d2	55-104 %R				1,1-Dichloroethene-d2	55-104 %R
		2-Butanone-d5	49-155 %R				2-Butanone-d5	49-155 %R
		Chloroform-d	78-121 %R				Chloroform-d	78-121 %R
		1,2-Dichloroethane-d4	78-129 %R				1,2-Dichloroethane-d4	78-129 %R
		Benzene-d6	77-124 %R				Benzene-d6	77-124 %R
		1,2-Dichloropropane-d6	79-124 %R				1,2-Dichloropropane-d6	79-124 %R
		Toluene-d8	77-121 %R				Toluene-d8	77-121 %R
		trans-1,3-Dichloropropene-d4	73-121 %R				trans-1,3-Dichloropropene-d4	73-121 %R
		2-Hexanone-d5	28-135 %R				2-Hexanone-d5	28-135 %R
		1,4-Dioxane-d8	50-150 %R				1,4-Dioxane-d8	50-150 %R
		1,1,2,2-Tetrachloroethane-d2	73-125 %R				1,1,2,2-Tetrachloroethane-d2	73-125 %R
		1,2-Dichlorobenzene-d4	80-131 %R				1,2-Dichlorobenzene-d4	80-131 %R
Internal Standards	all samples	60-140%		Check calculations and instruments, reanalyze affected samples	Karl Anderson, PACE	Accuracy	± 40 % of response area, ± 20 sec retention time shift	

QAPP Worksheet #28-C – Laboratory QC Samples Table for SVOCs

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

Matrix	**Aqueous/Soil/Waste**
Analytical Group	SVOCs
Concentration Level	Low to Medium
Sampling SOP	SESCO SOPs
Analytical Method/ SOP Reference	Refer to Work Sheet #19
Sampler's Name	TBD from SESCO and ER staff
Field Sampling Organization	SESCO/ER
Analytical Organization	Pace Analytical
No. of Sample Locations	Refer to Work Sheet #18

Lab QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
LCS/LCSD	1 every 12 hours	No analyte > RQL	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy Precision	Compound specific %R or %RSD
Calibration	As dictated by the method	SW-846, Method 8270D/ No analyte > RQL	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy	Factor of two(-50% to + 100%) from the initial/continuing calibration
Internal Standards	As dictated by the method	SW-846, Method 8270D	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy Precision	Compound specific %R or %RSD

Matrix Spike/Matrix Spike Duplicate	1 per ≤ 20 samples; if requested	SW-846, Method 8270D	Flag outliers	Karl Anderson, PACE	Accuracy Precision	Compound specific %R or %RSD
Surrogate Compounds	1 per < 20 samples; if requested	SW-846, Method 8270D	Flag outliers	Karl Anderson, PACE	Accuracy	Compound specific
Method Blank	As dictated by the method	SW-846, Method 8270D	Flag outliers	Karl Anderson, PACE	Accuracy	No analyte <RQL

QAPP Worksheet #28-D – Laboratory QC Samples Table for Pesticides/PCBs

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous/Soil/Waste
Analytical Group	Pesticides/PCB
Concentration Level	Low to Medium
Sampling SOP	SESCO SOPs
Analytical Method/ SOP Reference	Refer to Work Sheet #19
Sampler's Name	TBD from SESCO and ER staff
Field Sampling Organization	SESCO/ER
Analytical Organization	Pace Analytical
No. of Sample Locations	Refer to Work Sheet #18

Lab QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	1 every 12 hours	No analyte > RQL	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy Precision	Compound specific %R or %RSD
Calibration	As dictated by the method	SW-846, Method 8270D/ No analyte > RQL	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy	+/- 15%
Matrix Spike/Matrix Spike Duplicate	1 per ≤ 20 samples; if requested	SW-846, Method 8270D	Flag outliers	Karl Anderson, PACE	Accuracy Precision	Compound specific %R or %RSD
Surrogate Compounds	1 per < 20 samples; if requested	SW-846, Method 8270D	Flag outliers	Karl Anderson, PACE	Accuracy	Limits 30%-150%
Method Blank	As dictated by the method	SW-846, Method 8270D	Flag outliers	Karl Anderson, PACE	Accuracy	No analyte <RQL

QAPP Worksheet #28-E – Laboratory QC Samples Table for Metals/Mercury/Hexavalent Chromium

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous/Soil Matrix
Analytical Group	Metals/Mercury
Concentration Level	Low to Medium
Sampling SOP	SESCO SOPs
Analytical Method/SOP Reference	Refer to Work Sheet #19
Samplers Name	TBD from SESCO and ER Staff
Field Sampling Organization	SESCO/ER
Analytical Organization	Pace Analytical
No. of Sample Locations	Refer to Work Sheet #18

Lab QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	1 every 12 hours	SW-846, Method 6010C/7471/ 7196A	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy	Limits: Average Recovery ± 20%
Interference Check Sample (ICP/AES)	As dictated by the method	SW-846, Method 6010C/7471/ 7196A	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy	Within ± 2 times CRQL of true value or ± 20% of true value, whichever is greater, except for Al, Fe, Ca, K, Mg and Na
Preparation Blank	As dictated by the method	SW-846, Method 6010C/7471/ 7196A	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy	No analyte <RQL
Matrix Spike/Matrix Spike Duplicate	1 per ≤ 20 samples; if requested	SW-846, Method 6010C/7471/ 7196A	Flag outliers	Karl Anderson, PACE	Accuracy Precision	75 – 125 %R < 20 %RPD

Duplicate	1 per 10 samples; if requested	SW-846, Method 6010C/7471/ 7196A	Flag outliers	Karl Anderson, PACE	Precision	RPD < 20%
Serial Dilution Test (ICP/AES)	1 per < 20 samples; if requested	SW-846, Method 6010C/7471/ 7196A	Flag outliers	Karl Anderson, PACE	Accuracy	%D < 10 %
Instrument Calibration	As dictated by the method	SW-846, Method 6010C/7471/ 7196A	Flag outliers	Karl Anderson, PACE	Accuracy	90-100%
Post-Digestion Spike	As dictated by the method	SW-846, Method 6010C/7471/ 7196A	Flag outliers	Karl Anderson, PACE	Accuracy	Limits: Average Recovery ± 20%
Internal Standard (ICP-MS only)	As dictated by the method	SW-846, Method 6010C/7471/ 7196A	Flag outliers	Karl Anderson, PACE	Accuracy	Limits: 60 -125% RI

QAPP Worksheet #28-F – Laboratory QC Samples Table for PAHs

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous/Soil/Waste
Analytical Group	PAHs
Concentration Level	Low to Medium
Sampling SOP	SESCO SOPs
Analytical Method/ SOP Reference	Refer to Work Sheet #19
Sampler's Name	TBD from SESCO and ER staff
Field Sampling Organization	SESCO/ER
Analytical Organization	Pace Analytical
No. of Sample Locations	Refer to Work Sheet #18

Lab QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	1 every 12 hours	SW-846, Method 8270 SIM	Suspend analysis unit source recertified	Karl Anderson, PACE	Accuracy Precision	Compound specific %R
Matrix Spike/Matrix Spike Duplicate	1 per ≤ 20 samples; if requested	SW-846, Method 8270 SIM	Flag outliers	Karl Anderson, PACE	Accuracy Precision	Compound specific %R or %RSD
Surrogate Compounds	1 per < 20 samples; if requested	SW-846, Method 8270 SIM	Flag outliers	Karl Anderson, PACE	Accuracy	50 -150%R

Method Blank	As dictated by the method	SW-846, Method 8270 SIM	Flag outliers	Karl Anderson, PACE	Accuracy	No analyte <RQL Bis(2-ethylhexyl)phthalate < 5x CRQL

QAPP Worksheet #28-G – Laboratory QC Samples Table for TCLP

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

Matrix	Aqueous/Soil/Waste
Analytical Group	TCLP
Concentration Level	Low to Medium
Sampling SOP	SESCO SOPs
Analytical Method/ SOP Reference	Refer to Work Sheet #19
Sampler's Name	TBD from SESCO and ER staff
Field Sampling Organization	SESCO/ER
Analytical Organization	Pace Analytical
No. of Sample Locations	Refer to Work Sheet #18

Lab QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	As dictated by the method	SW-846, Method 1311	Flag outliers	Karl Anderson, PACE	Accuracy	Method dependent
Matrix Spike	1 per ≤ 20 samples; if requested	SW-846, Method 1311	Flag outliers	Karl Anderson, PACE	Accuracy	Method dependent
Extraction Time Limit	NA	SW-846, Method 1311	Flag outliers	Karl Anderson, PACE	Accuracy	VOC; N/A SVOC; 7 days Hg; N/A Metals; N/A

QAPP Worksheet #29 – Project Documents and Records Table

Identify the documents and records that will be generated for all aspects of the project including, but not limited to, sample collection and field measurement, on-site and off-site analysis, and data assessment.

☐ Worksheet Not Applicable (State Reason)

Document	Where Maintained
Field notes/logbook	Project file (SESCO)
Chain of custody forms	Project file (SESCO) and laboratory
Laboratory raw data package	Project file (SESCO)
Audit/assessment checklists/reports	Project file (SESCO) and laboratory
Corrective action forms/reports	Project file (SESCO) and laboratory
Laboratory equipment calibration logs	Laboratory
Sample preparation logs	Laboratory
Run logs	Laboratory
Sample disposal records	Laboratory
Validated data	Project file (SESCO)
Hard copy of analytical and raw data	Project file(SESCO)

QAPP Worksheet #30 – Analytical Services Table

Complete this worksheet for each matrix, analytical group, and concentration level. Identify all laboratories or organizations that will provide analytical services for the project, including on-site screening, on-site definitive, and off-site laboratory analytical work. If applicable, identify the subcontractor laboratories and backup laboratory or organization that will be used if the primary laboratory or organizations cannot be used.

☐ Worksheet Not Applicable (State Reason)

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory (Name and Address, Contact Person and Telephone Number)	Backup Laboratory (Name and Address, Contact Person and Telephone Number
Soil, water, liquid, solid	Metals, VOCs, SVOCs, PAHs, Pesticides, PCBs	Low to High	To be determined in the field	Refer to PACE Laboratories' SOPs, see QAPP Worksheet #23	Normal (21 days)	Pace Analytical Services 7726 Moller Road, Indianapolis, IN 46268 Karl Anderson (317) 875-5894	To be subcontracted for each project

QAPP Worksheet #31 – Planned Project Assessments Table

Identify the type, frequency, and responsible parties of planned assessment activities that will be performed for the project.

☐ Worksheet Not Applicable (State Reason)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Field Operation Audit	1 during Site activities	Internal	EPA OSC/START	Shelly Lam, OSC or START Contractor	SESCO Project Manager	SESCO Project Manager	EPA OSC
Laboratory Audit	TBD	External	SESCO chemist	TBD	SESCO Project Manager	TBD	TBD

QAPP Worksheet #32 – Assessment Findings and Corrective Action Responses

For each type of assessment describe procedures for handling QAPP and project deviations encountered during the planned project assessments.

☐ Worksheet Not Applicable (State Reason)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Laboratory Technical Systems/ Performance Audits	Audit Report	Shelly Lam, OSC	30 days	Letter	Pace Analytical	14 days
Laboratory Technical Systems/ Performance Audits	Audit Report	Shelly Lam, OSC, Laboratory	30 days	Letter	Pace Laboratory	14 days
Peer Review	Directly on deliverable	Shelly Lam, OSC	Prior to deliverable due date	Comments directly on deliverable	SESCO Project Manager	Prior to deliverable due date
Field Observations	Logbook	Shelly Lam, OSC, SESCO project manager	Immediately	Logbook	SESCO Team Leader	Immediately
Deviations from QAPP	Field Change Form	Shelly Lam, OSC	Immediately	Field Change Form	Project manager, SESCO	Within one business day of deviation

QAPP Worksheet #33 – QA Management Reports Table

Identify the frequency and type of planned QA Management Reports, the projected delivery date, the personnel responsible for report preparation, and the report recipients.

☐ Worksheet Not Applicable (State Reason)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Technical Report	Monthly	10th of the month following performance period	Project Manager/SESCO	Shelly Lam, OSC
QA Report	Quarterly	March, June, September	QA/QC Officer/SESCO	Shelly Lam, OSC

QAPP Worksheet #34 – Verification (Step I) Process Table

Describe the processes that will be followed to verify project data. Verification inputs include items such as those listed in Table 9 of the UFP-QAPP Manual. Describe how each item will be verified, when the activity will occur, and what documentation is necessary, and identify the persons responsible. *Internal* or *external* is in relation to the data generator.

☐ Worksheet Not Applicable (State Reason)

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain-of-custody 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 should 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. Copies of the chain-of-custody forms will be attached to the data report. See SOPs for further details.	Internal	SESCO Project Manager
Sample Receipt Forms	Sample receipt forms will be reviewed internally upon their completion and verified against the chain of custody forms. A copy of the sample receipt form will be retained in the project file, and the original included by the laboratory personnel in the data packages.	Internal	SESCO Project Manager
Field notes/logbook	Field notes will be reviewed internally and placed in the site file. A copy of the field notes will be attached to the final report.	Internal	SESCO Project Manager
Laboratory data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received data packages will be verified externally according to the data validation procedures specified in Worksheet #36.	Internal, External	SESCO Project Manager Laboratory
Laboratory EDDs / NEDDs	If required, laboratory EDDs/NEDDs will be verified against the laboratory data packages by the laboratory performing the work for completeness and technical accuracy prior to submittal. 10% of the received EDDs/NEDDs will be verified externally bySESCO.	Internal, External	SESCO Project Manager

QAPP Worksheet #35 – Validation (Steps IIa and IIb) Process Table

Describe the processes that will be followed to validate project data. Validation inputs include items such as those listed in Table 9 of the UFP-QAPP Manual. Describe how each item will be validated, when the activity will occur, and what documentation is necessary and identify the person responsible. Differentiate between steps IIa and IIb of validation.

☐ Worksheet Not Applicable (State Reason)

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Communication	Establish that required communication procedures were followed by field or laboratory personnel	SESCO Chemist
IIa	Sampling Methods and Procedures	Establish that the required sampling methods were used and that any deviations were noted. Ensure that the sampling procedures and field measurements met performance criteria and that any deviations were documented.	SESCO Project Manager
IIa	Holding Times	Ensure that samples were analyzed within holding times specified in method, procedure, or contract requirements. If holding times were not met, confirm that deviations were documented, that appropriate notifications were made as stated in the project-specific Statement of Work to the laboratory.	Laboratory SESCO Chemist
IIa	Analytes	Ensure that required lists of analytes were reported as specified in governing documents (i.e., method, procedure, or contract).	SESCO Chemist
IIa	Analytical Methods and Procedures	Establish that the required analytical methods were used and that any deviations were noted. Ensure that the QC sample met performance criteria and that any deviations were documented.	SESCO Chemist
IIa / IIb	Data Qualifiers	Determine that the laboratory data qualifiers were defined in the laboratory data packages and applied as specified.	SESCO Chemist
IIa	Field Transcription	Authenticate transcription accuracy of sampling data (i.e., from field logbook to report).	SESCO Chemist, project manager
IIb	Sampling Plan	Determine whether the sampling plan was executed as specified (i.e. the number, location, and type of field samples were collected and analyzed as specified in this QAPP.	SESCO Chemist START

[1] Compliance and Comparison from the IDQTF UFP-QAPP Manual, 2005

QAPP Worksheet #35 – Validation (Steps IIa and IIb) Process Table (Continued)

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIb	Sampling Procedures	Evaluate whether sampling procedures were followed with respect to equipment and proper sampling support (e.g., techniques, equipment, decontamination, volume, temperature, preservative, etc.)	Field Team Leader, SESCO
IIb	Co-located Field Duplicates	Compare results of co-located field duplicates with criteria established in the SAP (FSP/QAPP).	SESCO Chemist
IIb	Project Quantitation Limits	Determine that quantitation limits were achieved, as outlined in the SAP (FSP/QAPP) and that the laboratory successfully analyzed a standard at the quantitation limit (QL)	SESCO Chemist
IIb	Performance Criteria	Evaluate QC data against project-specific performance criteria in the SAP (FSP/QAPP), i.e., evaluate quality parameters beyond those outlines in the methods.	SESCO Chemist

[1] Compliance and Comparison from the IDQTF UFP-QAPP Manual, 2005

QAPP Worksheet #36 – Analytical Data Validation (Steps IIa and IIb) Summary Table

Identify the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data.

☐ Worksheet Not Applicable (State Reason)

Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb[1]	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa/IIb	Groundwater	Metals, VOCs, SVOCs, PCBs, pesticides	Low, medium	EPA's National Functional Guidelines for Data Validation of Organics EPA's National Functional Guidelines for Data Validation of Inorganics	Chemist, SESCO
IIa/IIb	Soil	Metals, VOCs, SVOCs, PCBs, pesticides	Low, medium	EPA's National Functional Guidelines for Data Validation of Organics EPA's National Functional Guidelines for Data Validation of Inorganics	Chemist, SESCO

[1] Compliance and Comparison from the IDQTF UFP-QAPP Manual, 2005

Refer to the following page for additional audit and data quality assessment activities

Assessment	On-Site Assessment/Audit (TSA)	Lab Assessment/Audit*	Data Quality Assessment (TSA)	Field instrument Doc. Assessment (TSA)	Data Validation
Qualified Person	QA/QC Coordinator or trained designee	Chemist	QA Manager/Qualified Personnel	Project Manager	Trained Chemist/ Qualified Personnel
Evaluation	• Calibration Records • COC Procedure • Sampling Procedure • Field Analytical Procedure • Training Records • T &D of Waste • Adherence to QAPP • Documentation	• Follow Lab Requirements • Consistent with Lab QAPP	• Lab Data Review • All data received • TAT achieved • Proper COC • Correct analytical method • Hold times not exceeded • QC sample results w/in limits • Review blank sample data	• Internal Calibration is complete and accurate • Standards are not expired • Duplicates are accurate • QC checks performed • QC checks w/in limits • Documentation complete • Calculations and entries are complete • COC maintained	• EPA National Functional Guidelines for Data Validation
Frequency	1 during Site activities	As directed by EPA	Waste disposal samples (completed under validation for all else)	Complete review each day of use as instrument is calibrated/operated	All Assessment Data
Report Requirement	Checklist and/or memo	Audit finding report	Memo of findings/section in report	Checklist	Validation Report
Expected Date:					

QAPP Worksheet #37 – Usability Assessment

Describe 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. Describe how data quality issues will be addressed and how limitations of the use of the data will be handled.

☐ Worksheet Not Applicable (State Reason)

The Data Usability Assessment will be performed by SESCO. Bill Pickard, Project Manager, will be responsible for information in the Usability Assessment. He will also be responsible for assigning task work to the individual task members who will be supporting the Data Usability Assessment. Note that the Data Usability Assessment will be conducted on verified data. After the Data Usability Assessment has been performed, the results of the Data Usability Assessment will be presented in the final project report. The following items will be assessed and conclusions drawn based on their results:

Precision

Precision is a measure of the reproducibility among a set of replicate results or the agreement among repeat observations made under the same conditions. *Analytical* precision is the measurement of the variability associated with duplicate or replicate analyses. *Total* precision is the measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate field samples and measures variability introduced by both the laboratory and field operations. Field duplicate samples and matrix duplicate spiked samples will be analyzed to assess field and analytical precision. The precision measurement is determined using the relative percent difference (RPD) between the duplicate sample results. The RPD is calculated according to the following formula:

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100\%$$

where: A = first duplicate concentration
B = second duplicate concentration

For this project, the parameters evaluated to assess precision are field and laboratory duplicate samples, and matrix spike/matrix spike duplicate (MS/MSD) samples. Goals for sample collection and analysis are set at 10% for field duplicate samples (soil) and 5% for MS/MSD samples for all applicable analyses, as listed in Worksheet # 20.

Results of all field and laboratory duplicates will be presented separately in tabular format for each analysis. For each duplicate pair, the RPD will be calculated for each analyte whose original and duplicate values are both greater than or equal to the quantitation limit. The RPDs will be checked against the measurement performance criteria presented on Worksheet #12 and Attachment A. The RPDs exceeding criteria will be identified on the tables. Additionally, the RPD of each analyte will be averaged across all duplicate pairs whose original and duplicate values are both greater than or equal to the quantitation limit, and the combined overall average RPD for each analysis will be calculated for the field and laboratory duplicates. A discussion will follow summarizing the results of the field and laboratory precision. Any conclusions about the precision of the analyses will be drawn and any limitations on the use of the data will be described.

QAPP Worksheet #37 – Usability Assessment (continued)

Accuracy

Accuracy is the degree of agreement between an analytical measurement and a reference accepted as a true value. A program of sample spiking will be conducted to evaluate laboratory accuracy. This program provides analysis of the MS and MSD samples, laboratory control spike (LCS), and surrogate standards. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy according to the following formula:

$$Percent\ \text{Recovery} = \frac{S - C}{T} \quad x \quad 100$$

where S = Measured spike sample concentration
C = Sample concentration
T = True or actual concentration of the spike

MS and MSD samples will be prepared and analyzed at a frequency of 5%. LCS or spike blanks are also analyzed at a frequency of 5%. Surrogate standards are added to every sample analyzed for organic constituents. Worksheet 12 and Attachment A present accuracy goals based on the percent recovery of matrix spike, LCS, and surrogate results.

Accuracy/Bias Contamination

Results for all laboratory method blanks and instrument blanks will be presented separately in tabular format for each analysis. The results for each analyte will be checked against the measurement performance criteria presented on Worksheet #12. Results for analytes that exceed criteria will be identified on the tables. A discussion will follow summarizing the results of the laboratory accuracy/bias. Conclusions about the accuracy/bias of the analyses based on contamination will be drawn and limitations on the use of the data will be described.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that they are intended to represent. For this project, representative data will be obtained through careful selection of sampling locations and analytical parameters. Representative data will also be obtained through proper collection and handling of samples to avoid interference and minimize contamination.

Representativeness of data will also be ensured through established field and laboratory procedures and their consistent application. To aid in evaluating of the representativeness of the sample results, field and laboratory blank samples, and background samples will be evaluated for the presence of contaminants. Data determined to be nonrepresentative, by comparison with existing data, will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

QAPP Worksheet #37 – Usability Assessment (continued)

Sensitivity

The method detection limit (MDL) is the minimum concentration of an analyte that can be reliably distinguished from background noise for a specific analytical method. The quantitation limit represents the lowest concentration of an analyte that can be accurately and reproducibly quantified in a given sample matrix. Project quantitation limit goals (PQLGs) are contractually specified maximum quantitation limits for a sample matrix and are typically several times the MDL to allow for matrix effects. PQLGs are set liberally to establish minimum criteria for laboratory performance; actual laboratory quantitation limits may be substantially lower.

Worksheet # 15 contains a comparison of the PQLGs for the selected analytical methods in comparison to the applicable regulatory criteria. The purpose of this comparison is to show that the selected analytical methods, and associated PQLGs, are capable of quantifying contaminants of concern at or below the applicable screening level. In comparing the PQLGs to PRGs, however, it is important to note that actual laboratory quantitation limits may be lower than PQLGs and that estimates of analyte concentrations down to MDLs can typically be provided in order to allow comparisons to screening levels that are below PQLGs.

Analytical results may be reported as estimated values if concentrations are less than PQLGs but greater than MDLs. The MDL for each analyte will be listed as the detection limit in the laboratory's EDD.

The field sample results for each analyte will be checked against the quantitation limits presented on Worksheet #15. Results for analytes that exceed criteria will be identified on the tables. A discussion will follow summarizing the results of the laboratory sensitivity. Conclusions about the sensitivity of the analyses will be drawn and limitations on the use of the data will be described.

Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this SAP, and when none of the QC criteria that affect data usability are exceeded. When all data validation is completed, the percent completeness value will be calculated by dividing the number of useable sample results by the total number of sample results planned for this investigation.

Comparability

Comparability expresses the confidence with which one data set can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data. Analytical methods selected for each of the project sites are consistent with the methods used during previous investigations at these sites.

QAPP Worksheet #37 – Usability Assessment (continued)

Reconciliation

Each of the PQOs presented on Worksheet #12 will be examined to determine if the objective was met. This examination will include a combined overall assessment of the results of each analysis pertinent to an objective. Each analysis will first be evaluated separately in terms of the major impacts observed from the Data Validation, Data Quality Indicators, and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Based on the quality determined, the usability of the data for each analysis will be determined. Based on the combined usability of the data from all analyses for an objective, it will be determined if the PQO was met and whether project action limits were exceeded. The final report will include a summary of all the points that went into the reconciliation of each objective. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described.

REFERENCES:

Indiana Department of Environmental Management, Remediation Closure Guide, March 22, 2012. Indiana Department of Environmental

Management, Remediation Program Guide, February 2012; Screening Tables Updated March 2015

Indiana Department of Environmental Management, Risk Integrated System of Closure Technical Guide – January 31, 2006 Appendix 1 (Revised May 1, 2009), Table A – Residential Closure Level

USEPA. EPA Using Data From Other Sources – A Checklist for Quality Concerns. May 2000.

USEPA. EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5. March 2001, Reissue May 2006.

USEPA. Guidance for Quality Assurance Project Plans. EPA 240-R-02-009. EPA QA/G-5. December 2002.

USEPA. Guidance for Choosing a Sampling Design for Environmental Data Collection. EPA 240/R- 02/005. EPA QA/G-5S. February 2006.

USEPA. Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review. EPA-540-R-08-01. June 2008.
www.epa.gov/superfund/programs/clp/guidance.htm#som

USEPA. Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use EPA- 540-R-08-005. January 2009.
www.epa.gov/superfund/programs/clp/guidance.htm#som

USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review. OSWER 9240.1-51. EPA 540-R-10-011. January 2010.
www.epa.gov/superfund/programs/clp/guidance.htm#som

USEPA. Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Review. EPA-540-R-11-016. September 2011.
www.epa.gov/superfund/programs/clp/guidance.htm#som

Attachment A: Method Precision and Accuracy Goals

Attachment A: Method Precision and Accuracy Goals

HACH 8000 COD

Compound Name	CAS No.	MDL	PQL	Units	Control Limits %Recovery	Control Limits RPD
COD	C-004	1.17	5	mg/L	80-120	25

EPA 245.2

Compound Name	CAS No.	MDL	PQL	Units	%Recovery	RPD
Mercury	7439-97-6	0.000066	0.002	mg/L	80-120	20

SM 4500 H+B

Compound Name	CAS No.	MDL	PQL	Units	%Recovery	RPD
pH	pH	1	1	S.U.	QC Check +/- 0.05 pH units	6

SM 2540D TSS

Compound Name	CAS No.	MDL	PQL	Units	%Recovery	RPD
Total Suspended Solids	C-009	5	10	mg/L	80-120	5

EPA 200.7

Compound Name	CAS No.	MDL	PQL	Units	%Recovery	RPD
Aluminum	7429-90-5	0.0179	0.2	mg/L	85-115	20
Arsenic	7440-38-2	0.00298	0.01	mg/L	85-115	20
Barium	7440-39-3	0.000309	0.01	mg/L	85-115	20
Cadmium	7440-43-9	0.000163	0.005	mg/L	85-115	20
Chromium	7440-47-3	0.000318	0.01	mg/L	85-115	20
Copper	7440-50-8	0.00111	0.01	mg/L	85-115	20
Iron	7439-89-6	0.00954	0.1	mg/L	85-115	20
Lead	7439-92-1	0.00145	0.015	mg/L	85-115	20
Manganese	7439-96-5	0.000566	0.015	mg/L	85-115	20
Nickel	7440-02-0	0.0012	0.04	mg/L	85-115	20
Selenium	7782-49-2	0.00374	0.04	mg/L	85-115	20
Silver	7440-22-4	0.000581	0.01	mg/L	85-115	20
Zinc	7440-66-6	0.00397	0.02	mg/L	85-115	20

Attachment B: Standard Operating Procedures/Practices

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP)
REFERENCE METHOD: EPA SW-846 METHOD 6010B

SOP NUMBER:	S-IN-I-019-rev.10
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-I-019-rev.09

APPROVAL

General Manager — September 27, 2012 Date

Quality Manager — September 26, 2012 Date

Department Manager — September 25, 2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of metals in aqueous and solid environmental samples while meeting the requirements specified in SW-846 method 6010B.

2. Summary of Method

2.1. Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2. This method describes multielement determinations by Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES). The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.

2.3. Background correction may be required to compensate for spectral interferences. Background is measured adjacent to analyte lines at a wavelength selected to be free of spectral interference and which reflects the same change in background intensity as occurs at the wavelength measured. Background correction is not required in cases of line broadening where a correction would actually degrade the analytical result.

3. Scope and Application

3.1. This method is applicable to the determination of most trace elements, including metals, in solution. Refer to Table 1 for the list of metals and reporting limits. Refer to the LIMS for method detection limits.

3.2. This method is applicable to groundwater, surface water, wastewater, extract, leachate, soil, sediment, sludge and other solid samples.

3.3. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of ICP systems and interpretation of ICP data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. **Spectral interferences:** Overlap of emission lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light can contribute to spectral interferences. These interferences can typically be minimized by careful selection of quantitation wavelengths, inter-element corrections, and background correction.

4.2. **Physical interferences:** Changes in sample viscosity, surface tension, or other effects associated with sample transport and nebulization can produce significant inaccuracies, especially in samples containing high concentrations of dissolved solids and acids. Dissolved solids may build up on the nebulizer tip, altering the sample flow rate and causing instrument drift. These effects can be minimized by sample dilution or use of a specially designed high-solids nebulizer.

4.3. **High Salt Concentrations:** high salt concentrations in sample digestates can cause signal suppression and confuse interference tests.

4.4. Chemical interferences: Molecular compound formation, ionization effects, and solute vaporization effects are typically not significant with ICP determinations. If observed, they can be minimized by careful selection of plasma and spectrometer operating parameters.

4.5. Memory interferences: Sample deposition on the nebulizer tubing, spray chamber, and plasma torch can cause apparent sample carryover. Memory interferences can be minimized by flushing the system with rinse blanks between samples. If memory interference is suspected for a sample, the sample must be re-analyzed after a sufficient rinse period.

5. Safety

5.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and must be handled with extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

5.2. Samples: Take precautions when handling samples. Samples must always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

5.3. Equipment: Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	250mL in plastic container	- HNO_3 to pH of <2 - Samples received at pH>2 must be preserved to pH<2 with HNO_3 and equilibrate for 24 hours before being prepared for analysis.	Ambient or 0°C to 6°C	Must be analyzed within 6 months of the collection date.
Aqueous - Dissolved	250mL in plastic container	- Filter; HNO_3 to pH<2	Ambient or 0°C to 6°C	Must be analyzed within 6 months of the collection date.
Solid	50 grams in glass or plastic container	- No chemical preservation	Ambient or 0°C to 6°C	Must be analyzed within 6 months of the collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
ICP-AES	Thermo-Fisher iCAP6500 or equivalent	Equipped with and autosampler and data system

8.2. General Supplies

Item	Vendor	Description
Volumetric Flasks	Class A	Various capacities
Volumetric Pipettors	Eppendorf or equivalent	Various sizes
Autosampler Vials	Environmental Express or equivalent	
Analytical Balance	Ohaus or eqivalent	Capable of weighing to 0.01g
Graduated Cylinders	Class A	Various capacities
pH strips	Fisher or equivalent	Full range

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Argon	High purity, liquefied
Nitric acid	Concentrated, trace metal analyzed or equivalent
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analytes and is used to spike MS/MSD sets..	Same solution can be used for the LCS and MS/MSD
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Yttrium
Interference Check Standards	Prepared to contain a known amount of interfering elements that will provide an accurate test of the interelement correction factors. If the ICP will display overcorrection as a negative number, the additional spiking with interfered elements is not necessary..	ICSA (ICSAB for BP Samples only)

9.2.3 Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Calibration Standards	SPEX; catalog #'s MIXSTD1-100; MIXSTD2-100; MIXSTD3-100; MIXSTD4-100; MIXSTD5-100; PLS19-2Y; CLSN2-2Y; PLTI9-2Y; CLAG2-2Y or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Calibration Standards	Refer to Section 9.2.3.2	Must be prepared fresh weekly	Same as stock standards
Stock ICV Standard	Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working ICV Standard	Refer to Section 9.2.3.4	Must be prepared fresh weekly	Same as stock standard
Working Second Source Spiking Solution	Refer to Section 9.2.3.5	Standard is good for 6 months from date of preparation.	Same as stock standard
Stock Interference Check Standard A	SPEX; catalog # INT-A1 or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Interference Check Standard A (ICSA)	Refer to Section 9.2.3.6	Must be prepared fresh weekly	Same as stock standards
Stock Interference Check Standard AB	SPEX; catalog #INT-A1, Mix-1B, Mix-2B, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Interference Check Standard AB (ICSAB)	Refer to Section 9.2.3.8	Must be prepared fresh weekly	Same as stock standards
Stock CRDL standard	CPI; catalog # 4400-132804, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working CRDL standard	Refer to Section 9.2.3.10	Must be prepared fresh weekly	Same as stock standards
Stock Internal Standard	SPEX; catalog # PLY2-2X; 1000mg/L yttrium or equivalent VHG; catalog # TLIN-500; 10,000mg/L lithium or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Internal Standard	Refer to Section 9.2.3.11	Must be prepared fresh weekly	Same as stock standards

9.2.3 Standard Preparation Procedures

9.2.3.1 Stock Calibration Standard Details

The following table shows the seven stock standard mixes that may be used to prepare the initial calibration and calibration check standards:

Analyte	Concentration (mg/L)
Catalog # MIXSTD1-100	
Lead	500
Selenium	200
Cadmium	150
Zinc	150
Manganese	100
Beryllium	50
Catalog # MIXSTD2-100	
Iron	10,000
Barium	100
Cobalt	100
Copper	100
Vanadium	100
Catalog # MIXSTD3-100 + silicon PLSI9-2Y	
Arsenic	500
Molybdenum	100
Silicon	100
Catalog #MIXSTD4-100	
Calcium	1000
Potassium	400
Aluminum	200
Sodium	200
Chromium	20
Nickel	20
Catalog #MIXSTD5-100	
Magnesium	1000
Antimony	200
Thallium	200
Boron	100
Silver	50
Mix #6(combines the following: CLSN2-2Y; PLTI9-2Y)	
Tin	1000
Titanium	1000
Mix #7- Catalog #CLAG2-2Y	
Silver	1000

9.2.3.2 Working Calibration Standards Preparation

Prepared fresh weekly and diluted from the stock standard mixes listed above, using a reagent water mixture that is 5% nitric acid and 2% hydrochloric acid unless otherwise noted.

Working Std. ID	Stock Standard	Vol. of Stock Std.	Final Volume
Calibration Std. Mix 1	MIXSTD1-100	2mL	100mL
Calibration Std. Mix 2	MIXSTD2-100	1mL	100mL
Calibration Std. Mix 3	MIXSTD3-100 Silicon PLSI9-2Y	2mL 0.8mL	100mL
Calibration Std. Mix 4	MIXSTD4-100	5mL	100mL
Calibration Std. Mix 5	MIXSTD5-100	2mL	100mL
Calibration Std. Mix 6	Tin CLSN2-2Y Titanium PLTI9-2Y	1mL 1mL	100mL
Calibration Std. Mix 7	Silver CLAG2-2Y	0.2mL	100mL in 10% HCl solution

9.2.3.3 Stock ICV Standard Details

The following table shows the concentrations of the stock standards purchased from Inorganic Ventures as three mixes:

Analyte	Concentration (mg/L)
Inorganic Ventures PA-STD1B	
Arsenic	200
Barium	200
Beryllium	200
Cadmium	200
Cobalt	200
Chromium	200
Copper	200
Manganese	200
Nickel	200
Phosphorus	200
Lead	200
Selenium	200
Thallium	200
Lithium	200
Strontium	200
Vanadium	200
Zinc	200
Inorganic Ventures PA-STD2B	
Silicon	1000
Boron	200
Molybdenum	200
Antimony	200
Tin	200
Titanium	200
Zirconium	200
Silver	100
Inorganic Ventures PA-STD3B	
Aluminum	2000
Calcium	2000
Iron	2000
Potassium	2000
Magnesium	2000
Sodium	2000

9.2.3.4 Working ICV Standard Preparation

Add 0.5mL of each Stock ICV Standard mix to a 100mL volumetric flask and dilute to volume with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

9.2.3.5 Working Second Source Spiking Solution

Add 25.0 mL of each Stock ICV Standard mix to a 100 mL volumetric flask and dilute to volume with reagent water solution that is 2% nitric acid.

9.2.3.6 Stock Interference Check Standard A (ICSA) Details

SPEX Interference Check Standard A (ICSA)	
Aluminum	5000
Calcium	5000
Magnesium	5000
Iron	2000

9.2.3.7 Working Interference Check Standard A (ICSA) Preparation

Dilute 10mL of the Stock ICSA Standard to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

9.2.3.8 Stock Interference Check Standard AB (ICSAB) Details

SPEX INT-A1	
Al, Ca, Mg	5000
Fe	2000
SPEX Mix 1B	
As, Ba, Be, B, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl, V, Zn	100
SPEX Mix 2B	
Mo, Sb, Sn, Ti	100
Ag	50
Si	500

9.2.3.9 Working Interference Check Standard AB (ICSAB) Preparation

Dilute 10mL of the stock INT-A1 standard and 0.5mLof the stock Mix 1B and Mix 2B to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

9.2.3.10 Stock Contract Required Detection Limit (CRDL) Standard Details

When specified by client or program requirements, a low-level check standard, also known as a CRDL standard, must be analyzed prior to sample analysis and at the end of each analytical batch to bracket the client samples. Acceptance limits for all target elements is 50-150% recovery. The composition of the CRDL standard is listed as follows:

Element	Conc. (ug/mL)	Element	Conc. (ug/mL)
Aluminum	100	Manganese	5
Antimony	5	Molybdenum	15
Arsenic	5	Nickel	20
Barium	1.5	Potassium	2500
Beryllium	5	Selenium	5
Boron	75	Silver	5
Cadmium	0.5	Sodium	1000
Calcium	250	Thallium	10
Chromium	10	Tin	35
Cobalt	10	Titanium	10
Copper	5	Vanadium	15
Iron	50	Zinc	20
Lead	3	Gold	10
Magnesium	250	Strontium	10

9.2.3.11 Working Contract Required Detection Limit (CRDL) Standard Preparation

Dilute 0.2mL of the stock CRDL solution to 100mL with a reagent water solution that is 5% nitric acid and 2% hydrochloric acid.

9.2.3.12 Working Internal Standard Preparation

Dilute 5mL of yttrium stock standard (1000mg/L) and 10mL of lithium stock standard (10,000mg/L) to 1L with a reagent water solution that is 2% nitric acid for a final concentration of 5mg/L of yttrium and 100mg/L of lithium.

10. Calibration

10.1. Initial Calibration: Calibrate the ICP each working day according to the instrument manufacturer's recommended procedures. Flush the system with the Calibration Blank solution prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. The calibration curve must consist of a minimum of a calibration blank and a standard.

10.2. Linear Calibration: Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.3. If a multi-point calibration is performed, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

10.4. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.5. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110% and the RSD of replicate readings must be <5%.

10.6. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

10.7. Initial Calibration Blank (ICB): The ICB consists of a reagent water solution that is 5% HNO_3 and 2% HCl. An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, another ICB may be analyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable. **For BP, the ICB must be evaluated. If the absolute value of a negative concentration exceeds twice the established MDL, the ICB is considered to be unacceptable. Samples associated with a failed ICB must be re-analyzed unless the concentration of the target analyte is greater than 10 times the absolute value of the ICB result.**

10.9 Continuing Calibration Verification (CCV): A CCV must be analyzed immediately following the ICB, after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The acceptable recovery range for the CCV is 90-110% and the RSD of replicate readings must be <5%.

10.10 CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

10.11 Contract Required Detection Limit (CRDL) Standard: When specified by client or program requirements, a CRDL standard must be analyzed prior to sample analysis and at the end of each analytical batch to bracket the associated samples. Acceptance limits for all target elements is 50-150% recovery. This standard is required for BP samples only.

10.12 CRDL Corrective action: BP Samples associated with a failed CRDL must be re-analyzed unless the concentration of the target has failed high, then the associated samples determined to be <RL are reportable

10.13 Interference Check Standard A (ICSA): An ICSA must be analyzed at the beginning of each analytical run. ICSA must be 80-120% of the true value for the elements in the mix. Non-ICSA elements must be within +/-2x the reporting limit.

10.14 ICSA Corrective Action: If the ICSA fails the acceptance criteria, another ICSA may be analyzed. If the second ICSA fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSA must be reanalyzed. **Exception:** If the ICSA is >120% for any element in the mix or if any non-ICSA element is >2x the reporting limit, indicating high bias, associated samples determined to be <RL are reportable.

10.15 Interference Check Standard AB (ICSAB): An ICSAB must be analyzed at the beginning of each analytical run. ICSAB must be 80-120% of the true value for the elements in the mix. **ICSAB is required for BP samples only.**

10.16 ICSAB Corrective Action: If the ICSAB fails the acceptance criteria, another ICSAB may be analyzed. If the second ICSAB fails, then a new calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICSAB must be reanalyzed. **Exception:** If the ICSAB is >120% for any element in the mix, indicating high bias, associated samples determined to be <RL are reportable.

10.17 Continuing Calibration Blank (CCB): The CCB consists of a reagent water solution that is 5% HNO_3 and 2% HCl. A CCB must be analyzed after every 10 samples following the CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable. **For BP, the CCB must be evaluated. If the absolute value of a negative concentration exceeds twice the established MDL, the CCB is considered to be unacceptable. Samples associated with a failed CCB must be re-analyzed unless the concentration of the target analyte is greater than 10 times the absolute value of the CCB result.**

11 Procedures

11.1 Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data document the selection criteria of background correction points; analytical dynamic ranges; the applicable equations, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis.

11.2 Configure the ICP per manufacturer's instructions and allow it to become thermally stable.

11.3 Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.

11.4 Establish initial calibration as described in Section 10.

11.5 Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration blank
Mix 1
Mix 2
Mix 3
Mix 4
Mix 5
Mix 6
Mix 7
ICV
ICB
CRDL (if required)
ICSA
ICSAB (if required)
Method blank
LCS
Client samples
CCV
CCB
Client samples
CRDL (if required)

ICSA (if required)
ICSAB (if required)
CCV
CCB

11.6 The instrument performs two replicate readings for each analysis and the average of the two readings is used to derive the concentration. For samples, the difference between the two readings must be ≤20% RSD for values that are >4x the reporting limit. If the RSD is >20% for values that are >4x the reporting limit, the sample must be reanalyzed.

11.7 Calculations for water samples are performed directly by the instrument software since initial sample aliquot and final digestate volumes are the same. If dilutions were performed, the appropriate factors must be applied to sample values.

11.8 The data system calculates the soil samples using the following equation:

$$C_s = \frac{C * V * D}{W}$$

Where: Cs = sample concentration (mg/kg, dry-weight basis)
C = concentration in extract (average of two readings taken)
V = volume of extract
D = dilution factor
W = dry weight of solid sample extracted (kg)

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

11.9 Samples with analyte concentrations above the upper linear range must be diluted and reanalyzed or the over range results must be qualified as estimated.

12 Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or boiling chips	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be less than reporting limits	Re-digest and reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Internal Standard	Yttrium	Automatically added to each sample, blank, and standard as part of the analysis.	No acceptance criteria – used to monitor interferences. 60-140%	No corrective action required. Sample may be analyzed at a dilution if interference is indicated.

12.2 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

12.5 Post-Digestion Spike Addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

12.6 Dilution test: If the analyte concentration is sufficiently high, minimally, a factor of 10 above the instrument detection limit after dilution, an analysis of a 1:5 dilution should agree within +/-10% of the original determination. If not, a chemical or physical interference effect should be suspected.

13 Method Performance

13.1 **Method Detection Limit (MDL) Study**: An MDL study must be conducted every 12 months for each matrix per instrument.

13.2 **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

13.3 **Linear Dynamic Range Study**: A linear dynamic range study must be conducted for each element by analyzing increasing concentrations of at least three, preferably five different concentration standards across the range. One of these should be near the upper limit of the range. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Samples determined to be above the upper range limit must be diluted and reanalyzed. New dynamic ranges should be determined whenever there is a significant change in instrument response. For those analytes that periodically approach the upper limit, the range should be checked every six months. Refer to Section 7.2.5.4 of Method 6010B for more information.

13.4 **Interelement Correction Factors** must be verified and updated every 6 months or when an instrumentation change occurs. Refer to Section 3.1 of Method 6010B for more information.

14 Method Modifications

14.1 Mixed standard solutions are purchased as certified standards.

14.2 Instrument conditions may vary from those stated in the method.

14.3 Calibration blanks are evaluated to the reporting limit and not to three times the IDL.

15 Pollution Prevention and Waste Management

15.1 Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

15.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

15.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

16.1 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Method 6010B.

16.2 Pace Analytical Quality Manual; latest revision.

16.3 TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1 Table 1: Target Metals and Reporting Limits

18 Revisions

Document Number	Reason for Change	Date
S-IN-I-019-rev.7	1. General: revised SOP references where applicable. 2. Table 7.1 (aqueous): changed time period to 24 hours per updated 40CFR part 136 (2007). 3. Table 12.1: added "or lab-generated limits" to LCS and MS limit description. 4. Table 12.1: added line in chart for internal standards with acceptance limits. 5. Table 12.1: added language to the ICSA requirements for non-ICSA elements (within +/-2X the RL). 6. New Section 11.10: added requirement for one serial dilution per batch for BP samples. 7. Section 10.3: added sentence about rerunning samples (to match other SOPs). 8. Section 10.4; added "preceding or" before "following" (to match other SOPs). 9. Added new section 10.6 to address BP requirement for calibration blanks. 10. Section 11.8: added corrective action when CCV fails RPD requirement. Also added BP requirement to monitor RPD limits for samples and the corrective action to rerun samples.	21Jan2008
S-IN-I-019-rev.8	1. Section 8.0: revised equipment and supplies section to a list from a table and added "or equivalent" to specific supply information. 2. Section 9.0: removed 1:1 acid references and added "or equivalent" to specific reagent information. 3. Table 9.2: changed Interference Check Standard to Interference Check Standard A (ICSA) and added ICSAB information 4. Table 9.3: Added "or equivalent" to specific catalog numbers, changed Stock and Working Inter-Element Standard to Stock and Working Interference Check Standard A (ICSA), and added Stock and Working Interference Check Standard AB (ICSAB). 5. Sections 9.2.3.7 and 9.2.3.8.: changed Stock and Working Inter-Element Correction Solution to Stock and Working Interference Check Standard A (ICSA). 6. Sections 9.2.3.11 and 9.2.3.12.: added Stock and Working Interference Check Standard AB (ICSAB). 7. Table 12.1: changed Inter-Element Correction Check to Interference Check Standard A (ICSA), added Interference Check Standard AB (ICSAB), and removed reference to lab-generated limits for LCS and MS/MSD.	08May2009

S-IN-I-019-rev.09	1. Section 2: streamlined summary of method 2. Section 3: removed SOP references and added a statement that weights, volumes, limits, etc… are subject to change. 3. Section 7: updated storage requirements, clarified hold time requirements and added requirements for dissolved metals. 4. Section 8: changed to a tabular format and updated information. Removed supplies relating to sample digestion. 5. Section 9: changed to a tabular format 6. Section 10: completely revised to clarify each QC sample and its corrective action, added calibration calculations and added Arsenic profiling criteria. 7. Section 11: removed table of ICP settings and added final concentration calculations. 8. Table 12.1: removed CCB, CRDL, ICSA, ICSAB and As profiling. Added RPD criteria for MS/MSD 9. Section 13: removed SOP references, added IDL information and expanded LDR information 10. Section 15: changed NELAC to TNI 11. Section 16: updated title of Attachment 1 12. Attachment 1: removed wavelengths and 200.7 information and update RLs.	23Jun2011
S-IN-I-019-rev.10	1. Section 3.1: added reference to MDLs. 2. Table 9.2: removed reference to Profiling Standard 3. Table 9.3: removed reference to Profiling Standard 4. Section 9.2.3: removed reference to Profiling Standard 5. Section 9.2.3.2: changed to a tabular format 6. Section 10: removed reference to Profiling Standard. 7. Section 11.9: added language that over range results can be reported if qualified as estimated. 8. Table 12.1: revised method blank corrective action. 9. Inserted new Method Modifications section.	19Sep2012

Table 1: Target Metals and Reporting Limits

Metals	Aqueous (µg/L)	Solid (mg/kg)
Aluminum - Al	1000	50
Antimony - Sb	6	2
Arsenic - As	10	2
Barium - Ba	100	2
Beryllium - Be	4	0.5
Boron - B	100	5
Cadmium - Cd	5	2
Calcium - Ca	1000	50
Chromium - Cr	10	2
Cobalt - Co	50	2
Copper - Cu	20	2
Iron – Fe	100	50
Lead – Pb	10	2
Magnesium – Mg	1000	50
Manganese – Mn	50	2
Molybdenum - Mo	50	2
Nickel – Ni	50	2
Potassium - K	1000	50
Selenium - Se	10	2
Silver – Ag	50	2
Sodium – Na	1000	50
Thallium - Tl	50	2
Tin – Sn	10	2
Titanium - Ti	50	2
Vanadium - V	50	2
Zinc – Zn	50	2

STANDARD OPERATING PROCEDURE

ACID DIGESTION OF AQUEOUS SAMPLES FOR ICP

REFERENCE METHOD: EPA SW-846 METHOD 3010

SOP NUMBER:	S-IN-I-030-rev.10
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-I-030-rev.9

LOCAL APPROVAL

General Manager — August 30, 2011 Date

Quality Manager — August 30, 2011 Date

Department Manager — August 30, 2011 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for acid-digesting aqueous samples for metals analysis while meeting the requirements specified in EPA method 3010. The digestates are then analyzed by inductively coupled plasma (ICP).

2. Summary of Method

2.1. A portion of sample is digested with nitric acid in a hot block digester. After addition of hydrochloric acid, the samples are digested further, cooled and then brought to volume with reagent water.

3. Scope and Application

3.1. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of metals digestion equipment. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Not applicable to this SOP.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and should be handled with extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

5.3. Equipment

Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

6. Definitions

6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	250mL in plastic container	- HNO_3 to pH of <2 - Samples received at pH>2 must be preserved to pH<2 with HNO_3 and be allowed to equilibrate for 24 hours before being prepared for analysis.	Ambient or 0°C to 6°C	Must be analyzed within 6 months of the collection date.
Aqueous - Dissolved	250mL in plastic container	- Filter; HNO_3 to pH<2	Ambient or 0°C to 6°C	Must be analyzed within 6 months of the collection date.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
Hot Block Digester	Environmental Express or equivalent	Adjustable and capable of maintaining a temperature of 90°C to 95°C.
Analytical Balance	Ohaus or eqivalent	Capable of weighing to 0.01g

8.2. General Supplies

Item	Vendor	Description
Volumetric Flasks	Class A	Various capacities
Volumetric Pipettors	Eppendorf or equivalent	Various sizes
Digestion Tubes	Environmental Express or equivalent	Volumetrically certified and contaminant free
Thermometer	Ever Safe or equivalent	Calibrated, used for monitoring Hot Block temperature
Plunger Filters	Environmental Express or equivalent	
Graduated Cylinders	Class A	Various capacities
pH strips	Fisher or equivalent	Full range

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Nitric acid	Concentrated, trace metal analyzed or equivalent
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.1 Standard Definitions and vendors

Standard	Description	Comments
Spiking Standard	This solution contains the target analytes and is generally prepared using a standard source secondary to the standards used for calibration.	Same solution can be used for the LCS and MS/MSD
Internal Standard	A solution added all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Yttrium

9.2.2. Storage Conditions

Table 9.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Spiking Standard	Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Spiking Standard	Refer to Section 9.2.3.1.	Expires 6 months from date of preparation.	Same as stock standard

9.2.3. Preparation Procedures

Table 9.3 – Stock Spiking Standard Details

Analyte	Concentration (mg/L)
Inorganic Ventures PA-STD1B	
Arsenic	200
Barium	200
Beryllium	200
Cadmium	200
Cobalt	200
Chromium	200
Copper	200
Manganese	200
Nickel	200
Phosphorus	200
Lead	200
Selenium	200
Thallium	200
Lithium	200
Strontium	200
Vanadium	200
Zinc	200
Inorganic Ventures PA-STD2B	
Silicon	1000
Boron	200
Molybdenum	200
Antimony	200
Tin	200
Titanium	200
Zirconium	200
Silver	100

Inorganic Ventures PA-STD3B	
Aluminum	2000
Calcium	2000
Iron	2000
Potassium	2000
Magnesium	2000
Sodium	2000

9.2.3.1. Working Spiking Standard Preparation

Dilute 50mL of each stock spiking standard (solutions 1B, 2B and 3B) and 4mL concentrated Nitric acid to 200mL with reagent water. One mL of this solution is used to spike the LCS, MS and MSD for 50mL initial volumes. For 15mL initial volumes use 0.3mL of this spike for LCS, MS and MSD.

10. Calibration

10.2. Not applicable to this SOP.

11. Procedures

11.1 If lower reporting limits are required, digestate concentration may be performed provided that final acid concentration and final spike concentration remain consistent with unconcentrated digestates.

11.2. Transfer 50mL of well-mixed sample into a digestion tube and add 2.5mL concentrated nitric acid. If using a 15 mL sample volume, add 0.75mL concentrated nitric acid. Place the sample into the Hot Block and set the temperature to achieve 95^{o}C (+/- 3^{o}C) in the digestion vessels.

11.3. Digest the sample until volume is below 10mL but not dry for 50mL sample volumes and to 3mL but not dry for 15mL sample volumes. Allow sample to cool.

11.4. Add another portion of nitric acid and continue heating on the Hot Block. If brown fumes are generated, add another portion of nitric acid and continue heating on the Hot Block until the digestate is light in color and no brown fumes are given off, indicating a complete digestion. Do not allow the digestate to go dry. Allow samples to cool.

11.5. Add 2.0mL of concentrated hydrochloric acid to each 50mL sample or 0.6mL concentrated hydrochloric acid to each 15mL sample and warm on the Hot Block for another 15 minutes in order to dissolve any precipitate. Remove samples from Hot Block and cool.

11.6. Dilute the digestate to the desired final volume in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered.

11.7. Record all digestion information including standard numbers, reagent numbers, digestion tube lot numbers, filter lot numbers, Hot Block number and digestion temperature in the metals digestion logbook and deliver the digestates to the ICP analyst..

12. Quality Control

12.2. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be less than reporting limits	Re-digest and reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, the sample data may be reported without qualifiers, the method blank must be qualified.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

12.3. Method Blank Preparation: the Method Blank consists of Reagent Water.

12.4. LCS Preparation: For 50mL initial volumes, add 1mL of the Working Spiking Standard to 50mL of reagent water. For 15mL initial volumes, add 0.3mL of the Working Spiking Standard to 15mL of reagent water. Spike concentration is 1mg/L.

12.5. Matrix Spike Preparation: For 50mL initial volumes, add 1mL of the Working Spiking Standard to 50mL of sample. For 15mL initial volumes, add 0.3mL of the Working Spiking Standard to 15mL of sample. Spike concentration is 1mg/L.

12.6. LCS calculation

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.7. MS/MSD calculation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.8 RPD calculation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13. Method Performance

13.2. Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

13.3. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14. Pollution Prevention and Waste Management

14.2. Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP.

14.3. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.4. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. References

15.2. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Methods 3010 and 3020.

15.3. Pace Analytical Quality Manual; latest revision.

15.4. TNI Standard; Quality Systems section; latest revision.

16. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

16.1. Not applicable to this SOP.

17. Revisions

Document Number	Reason for Change	Date
S-IN-I-030-rev.7	1. Table 12.1: changed batch time from 12 hours to 8 hours. 2. General: revised SOP references where applicable. 3. Added new section 11.3 to show that sample needs to have continued addition of nitric acid until digestion is complete.	21Jan2008
S-IN-I-030-rev.8	1. Cover sheet: revised to include annual review signatures and removed GFAA references. 2. Sections 1 and 2: removed GFAA references. 3. Table 7.1: revised collection volume, expanded procedures when samples are preserved by the lab and added refrigeration as an acceptable form of storage. 4. Section 8: changed tables to lists and removed vendor information. 5. Table 9.3: removed GFAA references. 6. Section 9.2.3.3: removed because of GFAA references. 7. Sections 11.4 and 11.6: removed GFAA references.	22Apr2009
S-IN-I-030-rev.9	1. Table of Contents: fixed to include Sections 10 and 11 2. Table 9.2: changed working standard expiration to 6 months 3. Section 9.2.3.1: revised to include spike amounts for 15mL samples 4. Section 11.1: added allowances for digestate concentration to achieve lower reporting limits 5. Sections 11.2, 11.3 and 11.5: added procedure for optional 15mL sample volume 6. Section 11.6: added requirement for filtration of method blank when digestates are filtered. 7. Table 12.1: revised to include spike amounts for 15mL sample volume and exception for method blank rejection.	15Jan201
S-IN-I-030-rev.10	1. Cover page revised to most recent format. 2. Table 7.1: Removed TCLP-specific information, added information for handling of dissolved metals, revised storage requirements and volume required. 3. Section 8: converted to a tabular format and updated information. 4. Section 9: converted to a tabular format and updated information. 5. Section 11.2: added “well-mixed” 6. Section 11.4: added details for handling of samples that give off brown fumes during digestion. 7. Section 11.5: updated acid volumes added. 8. Section 11.6: added LCS after method blank in last sentence. 9. Section 11.7: added Hot Block number and digestion temperature as information to be recorded. 10. Section 12: revised Table 12.1 and added preparation instructions for Method Blank, LCS and Matrix Spike. 11. Section 13: removed SOP references. 12. Section 15: changed NELAC to TNI.	30Aug2011

STANDARD OPERATING PROCEDURE

ACID DIGESTION OF SOLID SAMPLES FOR ICP ANALYSIS

REFERENCE METHOD: EPA SW-846 METHOD 3050B

SOP NUMBER:	S-IN-I-031-rev.10
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-I-031-rev.9

APPROVAL

Karl Anderson

General Manager — August 30, 2011 — Date

Quality Manager — August 30, 2011 — Date

David D. Mark

Department Manager — August 30, 2011 — Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for acid-digesting solid samples and wipes for metals analysis while meeting the requirements specified in EPA method 3050B. The digestates are then analyzed by inductively coupled plasma (ICP).

2. Summary of Method

2.1. A sample is digested with nitric acid and hydrogen peroxide in a hot block digester. After the addition of hydrochloric acid, the samples are digested further, cooled, then brought to volume with reagent water.

3. Scope and Application

3.1. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of metals digestion equipment. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Not applicable to this SOP.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock metals standards are toxic and should be handled with extreme care. Also handle concentrated acids with care, making sure to wear appropriate personal protective equipment.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

5.3. Equipment

Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

6. Definitions

6.1 Refer to Glossary section of the Pace Quality Assurance Manual for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	50 grams in glass or plastic container	No chemical preservation required	0°C to 6°C	Must be analyzed within 6 months of the collection date.
Wipe	Individual wipe in glass or plastic container	No chemical preservation required	0°C to 6°C	Must be analyzed within 6 months of the collection date.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
Hot Block Digester	Environmental Express or equivalent	Adjustable and capable of maintaining a temperature of 90°C to 95°C.
Analytical Balance	Ohaus or eqivalent	Capable of weighing to 0.01g

8.2. General Supplies

Item	Vendor	Description
Volumetric Flasks	Class A	Various capacities
Volumetric Pipettors	Eppendorf or equivalent	Various sizes
Digestion Tubes	Environmental Express or equivalent	Volumetrically certified and contaminant free
Teflon Chips	Fisher or equivalent	For use as a clean solid matrix
Thermometer	Ever Safe or equivalent	Calibrated, used for monitoring Hot Block temperature
Plunger Filters	Environmental Express or equivalent	
Graduated Cylinders	Class A	Various capacities
pH strips	Fisher or equivalent	Full range
Ghost Wipes	Environmental Express or equivalent	Mixed cellulose ester wipe

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Nitric acid	Concentrated, trace metal analyzed or equivalent
Hydrochloric acid	Concentrated, trace metal analyzed or equivalent
Hydrogen Peroxide	30% solution, trace metal analyzed or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

Standard	Description	Comments
Spiking Standard	This solution contains the target analytes and is generally prepared using a standard source secondary to the standards used for calibration.	Same solution can be used for the LCS and MS/MSD
Internal Standard	A solution added all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	Yttrium

9.2.2. Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Spiking Standard	Inorganic Ventures; catalog #s PA-STD-1B; PA-STD-2B; PA-STD-3B, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working Spiking Standard	Refer to Section 9.2.3.1.	Expires 6 months from date of preparation.	Same as stock standard

Table 9.3 – Analytical Standard Storage Conditions

9.2.3. Preparation Procedures

Table 9.3 - Stock Spiking Standard Details

Analyte	Concentration (mg/L)
Inorganic Ventures PA-STD1B	
Arsenic	200
Barium	200
Beryllium	200
Cadmium	200
Cobalt	200
Chromium	200
Copper	200
Manganese	200
Nickel	200
Phosphorus	200
Lead	200
Selenium	200
Thallium	200
Lithium	200
Strontium	200
Vanadium	200
Zinc	200
Inorganic Ventures PA-STD2B	
Silicon	1000
Boron	200
Molybdenum	200

Antimony	200
Tin	200
Titanium	200
Zirconium	200
Silver	100
Inorganic Ventures PA-STD3B	
Aluminum	2000
Calcium	2000
Iron	2000
Potassium	2000
Magnesium	2000
Sodium	2000

9.2.3.1. Working Spiking Standard Preparation

Dilute 50mL of each stock spiking standard (solutions 1B, 2B and 3B) and 4mL concentrated Nitric acid to 200mL with reagent water. One mL of this solution is used to spike the LCS, MS and MSD for 50mL initial volumes. For 15mL initial volumes use 0.3mL of this spike for LCS, MS and MSD.

10. Calibration

10.1. Not applicable to this SOP.

11. Procedures

11.1. Weigh 1g of well-mixed sample into a labeled digestion tube and add 5mL of reagent water and 5mL of concentrated nitric acid. Place the digestion tube into the Hot Block and set the temperature to achieve 95^{o}C +/- 3^{o}C in the digestion vessels. For ghost wipes, place the wipe in a labeled digestion tube and add 20mL of reagent water to completely cover the wipe. Add 5mL of concentrated nitric acid. Place the digestion tube into the Hot Block and set the temperature to achieve 95^{o}C +/- 3^{o}C in the digestion vessels.

11.2. Digest the sample until the volume is below 10mL but not dry. If brown fumes are generated, add 5mL of nitric acid and continue to digest, do not let the sample go dry. Continue adding nitric acid and digesting to below 10 mL until no brown fumes are given off, indicating a complete digestion.

11.3. Add 1mL of 30% hydrogen peroxide solution and warm slowly until effervescing stops. Continue to add 30% hydrogen peroxide solution in 1mL aliquots while warming until the effervescing is at a minimum or the general appearance of the sample is unchanged. Do not add more than 5mL total of the hydrogen peroxide solution.

11.4. Continue heating the acid –peroxide digestate until the volume has been reduced to approximately 5mL. Do not allow the digestate to go to dryness.

11.5. Allow the samples to cool. Add 5mL concentrated hydrochloric acid to each sample and warm the samples for another 15 minutes in order to dissolve any precipitate.

11.6. Adjust the volume to 50mL in the digestion tube with reagent water. Filter the samples to remove particulates using a plunger filter. The associated Method Blank and LCS must also be filtered.

11.7. Record all information including standard numbers, reagent numbers, digestion tube lot numbers, filter lot numbers, Hot Block number and digestion temperature in the metals digestion logbook and deliver the digestates to the ICP analyst.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	1g Teflon chips for solid samples or one unused ghost wipe for wipe samples	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be less than reporting limits	Re-digest and reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, the sample data may be reported without qualifiers, the method blank must be qualified.
Laboratory Control Sample (LCS)	Applicable target analyte + 1g Teflon chips for solid samples or one unused ghost wipe for wipe samples	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte + sample	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

12.2 Method Blank Preparation: For the Method Blank, add 1g of Teflon chips or one unused ghost wipe.

12.3 LCS Preparation: For LCS, add 1mL of the Working Spiking Standard plus 1g of Teflon chips or one unused ghost wipe.

12.4 Matrix Spike Preparation: Add 1mL of the Working Spiking Standard plus 1g of sample.

12.5 LCS calculation:

R = (C/S) * 100

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.6 MS/MSD calculation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.7 RPD calculation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

12.8 Wipe Calculation:

Total ug analyte per wipe = (analyte conc., ug/L) x (0.050 Liter/wipe)

13 Method Performance

13.2 Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

13.3 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14 Pollution Prevention and Waste Management

14.2 Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP.

14.3 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.4 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15 References

15.2 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, latest revision, Method 3050B.

15.3 Pace Analytical Quality Manual; latest revision.

15.4 TNI Standard; Quality Systems section; latest revision.

16 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

16.2 Not applicable to this SOP.

17 Revisions

Document Number	Reason for Change	Date
S-IN-I-031-rev.8	1. Table 8.2, Table 12.1 and section 9.2.3.2: added wording about solid LCS material. 2. Table 12.1: changed batch time from 12 hours to 8 hours. 3. General: updated SOP references where applicable. Referenced 3050B as method revision. 4. New section 11.2 added describing the process of additional nitric acid additions until no brown fumes are generated.	21Jan2008
S-IN-I-031-rev.9	1. Table of Contents: fixed to include Sections 10 and 11. 2. Sections 8 and 9: revised to be in list form and removed catalog number references. 3. Section 9.2.3.2: reworded for clarity. 4. Table 9.3: changed working standard expiration to 6 months. 5. Table 12.1: added 10x rule exception for Method Blank corrective action.	04Jan2010
S-IN-I-031-rev.10	1. Cover page revised to most recent format. 2. Table 7.1: revised storage requirements and volume required and added wipes. 3. Section 8: converted to a tabular format and updated information, added wipes. 4. Section 9: converted to a tabular format and updated information. 5. Section 11.1: added "well-mixed" and procedure for wipes 6. Section 11.3: updated peroxide volumes added. 7. Section 11.6: added LCS after method blank in last sentence. 8. Section 11.7: added Hot Block number and digestion temperature as information to be recorded. 9. Section 12: revised Table 12.1 and added preparation instructions for Method Blank, LCS and Matrix Spike. 10. Section 13: removed SOP references. 11. Section 15: changed NELAC to TNI.	30Aug2011

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF MERCURY BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY

REFERENCE METHODS: EPA SW-846 METHODS 7470A AND 7471A

SOP NUMBER:	S-IN-I-040-rev.13
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-I-040-rev.12

APPROVAL

General Manager	November 2, 2012 Date
Quality Manager	October 30, 2012 Date
Department Manager	October 29, 2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
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1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining total mercury concentration while meeting the requirements specified in EPA method 7470A for aqueous samples and method 7471A for solid samples.

2. Summary of Method

2.1. Prior to analysis, all samples are digested by heating with appropriate acids and oxidizing agents to dissolve and oxidize mercury contents.

2.2. This cold-vapor method is based on the absorption of radiation at 253.7nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

3. Scope and Application

3.1. This method is applicable for the measurement of mercury in groundwater, surface and saline waters, domestic and industrial wastes, TCLP extracts, soil, sediment, bottom deposits and sludge-type materials.

3.2. The reporting limits for mercury are 0.002 mg/L for aqueous samples and 0.33 mg/Kg for solid samples. Refer to the LIMS for method detection limits.

3.3. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of mercury analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. High concentrations of sulfide may interfere in some water or solid samples. Potassium permanganate is added during digestion to eliminate sulfide interference. Concentrations as high as 20mg/L in water or 20mg/kg in soils have been demonstrated to cause no interference in spiked samples.

4.2. High concentrations of copper have been reported to interfere with mercury determinations. Concentrations as high as 10mg/L in water or 10mg/kg in soil have been demonstrated to cause no interference in spiked samples.

4.3. High concentrations of chloride, present in samples require additional potassium permanganate. The free chlorine produced during digestion should be removed with excess hydroxylamine hydrochloride solution.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. The use of gloves, lab coats and safety glasses is required. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous - Total	500 mL in plastic container.	HNO_3 to pH<2 Samples received at pH>2 must be preserved to pH<2 with HNO_3 and be allowed to equilibrate for 24 hours before being prepared for analysis.	Ambient	Analysis must be completed within 28 days of collection date.
Aqueous - Dissolved	500 mL in plastic container	Filter; HNO_3 to pH<2	Ambient	Analysis must be completed within 28 days of collection date.
Solid	100g in a 4oz glass container	None	0°C to 6°C	Analysis must be completed within 28 days of collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Automated Mercury Analyzer	CETAC	M-7500 or equivalent	To include an atomic absorption spectrophotometer, mercury lamp, absorption cell, air pump, flow meter, drying tube, autosampler and data system.
Hot Block	Environmental Express	56-well or equivalent	Adjustable and capable of maintaining a temperature of 90°C to 95°C.
Balance	OHaus	GT400 or equivalent	Readability to 0.01g

8.2. General Supplies

Item	Vendor	Description
Auto-pipettes	Eppendorf or equivalent	Various sizes
Volumetric flasks	Class A	100mL
Graduated cylinder	Class A	25mL
Digestion cups	Environmental Express or equivalent	50mL capacity, volumetrically certified
Autosampler tubes	Moldpro, Inc or equivalent	17x100 mm

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Sulfuric acid	Concentrated, trace metal grade or equivalent
Hydrochloric acid	Concentrated, trace metal grade or equivalent
Hydrochloric acid (3%)	Dilute 30 mLs of concentrated HCl to 1L with reagent water. Used for dilution preparation.
Nitric acid	Concentrated, trace metal grade or equivalent
Aqua Regia	Carefully add one volume of nitric acid to three volumes of hydrochloric acid. Must be prepared in a hood and must be prepared immediately before use on each day.
Stannous Chloride solution	Add 100g stannous chloride and 70 mL conc. HCl in reagent water and dilute to 1L. This solution is good for 3 days. Refrigerate when not in use.
Sodium Chloride	Granular
Hydroxylamine hydrochloride	Granular
Sodium Chloride/ Hydroxylamine Hydrochloride solution	Dissolve 120g sodium chloride and 120g hydroxylamine hydrochloride in reagent water and dilute to 1L. This solution is good for 6 months from preparation (hydroxylamine sulfate may be substituted for hydroxylamine hydrochloride).
Potassium permanganate solution (5%)	Commercially purchased Mercury-free, 5% solution (w/v)
Potassium persulfate	Granular
Potassium persulfate solution	Dissolve 5g Potassium persulfate in reagent water and dilute to 100mL.
Rinse/Probe Wash Solution	Add 50mL HCL and 20mL HNO3 to 500mL reagent water and dilute to 1L.
Boiling chips	Or equivalent to be used as a simulated soil matrix.

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Reporting Limit Verification Standard	A standard prepared at a concentration equivalent to the reporting limit for verification at that level.	RLVS
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains the target analyte and is used to spike MS/MSD sets.	Same solution can be used for both the LCS and MS/MSD.

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Mercury Calibration standard	Fisher; catalog # SM114-500; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate #1 Mercury Calibration standard	Refer to Section 9.2.3.1	Solution expires 6 months from date of preparation.	Same as for stock standard.
Intermediate #2 Mercury Calibration standard	Refer to Section 9.2.3.1	Must be prepared fresh daily.	Not Applicable
Working Mercury Calibration standards	Refer to Section 9.2.3.2	One-time use standards.	Not Applicable
Stock Mercury ICV/Spiking standard	SPEX; catalog # PLHG4-2Y; 1000mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate #1 Mercury ICV/Spiking standard	Refer to Section 9.2.3.3	Solution expires 6 months from date of preparation.	Same as for stock standard.
Intermediate #2 Mercury ICV/Spiking standard	Refer to Section 9.2.3.3	Must be prepared fresh daily.	Not Applicable
Working Mercury ICV standard	Refer to Section 9.2.3.4	One-time use standard.	Not Applicable
Working RLVS standard	Refer to Section 9.2.3.2	One-time use standard.	Not Applicable

9.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for specific instructions regarding preparation of standards for Mercury analysis

9.2.3.1 Intermediate Mercury Calibration Standard Preparation

Intermediate #1 Mercury Calibration Standard: Dilute 1mL of the Stock Mercury Calibration Standard (1000mg/L) to 100mLs with 2% HNO_3 for a final concentration of 10mg/L. This standard is good for 6 months from the date of preparation.

Intermediate #2 Mercury Calibration Standard: Dilute 1mL of the Intermediate #1 Mercury Calibration Standard (10mg/L) to 100mLs with 2% HNO_3 for a final concentration of 100ug/L. This standard must be prepared fresh daily.

9.2.3.2 Working Mercury Calibration Standards Preparation

Working calibration standards are one-time use and are prepared by diluting the Intermediate #2 Mercury Calibration Standard with reagent water. Examples of possible calibration standards are as follows:

Aqueous:

Standard ID	Amount of Int. Std. #2	Final Volume in reagent water	Final Concentration
Standard 1 (RLVS)	0.06mL	30mL	0.2ug/L
Standard 2	0.3mL	30mL	1.0ug/L
Standard 3	0.6mL	30mL	2.0ug/L
Standard 4 (CCV)	1.5mL	30mL	5.0ug/L
Standard 5	2.25mL	30mL	7.5ug/L
Standard 6	3.0mL	30mL	10.0ug/L

Solid:

Standard ID	Amount of Int. Std. #2	Final Volume in reagent water	Final Concentration
Standard 1 (RLVS)	0.1mL	50mL	0.2ug/L
Standard 2	0.5mL	50mL	1.0ug/L
Standard 3	1.0mL	50mL	2.0ug/L
Standard 4 (CCV)	2.5mL	50mL	5.0ug/L
Standard 5	3.75mL	50mL	7.5ug/L
Standard 6	5.0mL	50mL	10.0ug/L

9.2.3.3 Intermediate Mercury ICV/Spiking Standard Preparation

Intermediate #1Mercury ICV Standard: Dilute 1mL of the Stock Mercury ICV/Spiking Standard (1000mg/L) to 100mLs with 2% HNO_3 for a final concentration of 10mg/L. This standard is good for 6 months from the date of preparation.

Intermediate #2Mercury ICV Standard: Dilute 1mL of the Intermediate #1 Mercury ICV/Spiking Standard (10mg/L) to 100mLs with 2% HNO_3 for a final concentration of 100ug/L. This standard must be prepared fresh daily and is also used to prepare the LCS and MS/MSD.

9.2.3.4 Working Mercury ICV/LCS Standard Preparation

Aqueous: Dilute 1.5mL of the Intermediate #2 Mercury ICV Standard to 30mL with reagent water for a standard concentration of 5.0ug/L. This standard is a one-time use standard and is also used as the LCS.

Solid: Dilute 1.5mL of the Intermediate #2 Mercury ICV Standard to 50mL with reagent water for a standard concentration of 3.0ug/L. This standard is a one-time use standard and is also used as the LCS.

10. Calibration

10.1. Initial Calibration: A minimum of 5 calibration standards is required. The lowest calibration standard must be at or below the reporting limit. A new initial calibration curve with freshly prepared standard is analyzed on each working day. Refer to the Quality Manual for more information regarding calibration curves.

10.2. Linear Calibration: Using the instrumentation software, prepare a standard curve by plotting absorbance versus mercury concentration of each calibration standard. The analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.3. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

$$r = \frac{\sum_{i=1}^{N} (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} (Y_i - \overline{Y})^2\right)}}$$

10.4. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be digested analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed. Refer to Section 10.13 for additional information.

10.5. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

10.6. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported. Refer to Section 10.13 for additional information.

10.7. Initial Calibration Blank (ICB): The ICB consists of reagent water that is prepared per Section 11. An ICB must be analyzed immediately following the ICV. If the ICB result is above the reporting limit, the ICB may be reanalyzed. If the second ICB fails, then a new initial calibration curve must be analyzed. Samples associated with a failed ICB must be reanalyzed. **Exception:** If the ICB is >RL, associated samples determined to be <RL are reportable. Refer to Section 10.13 for additional information.

10.8. Continuing Calibration Verification (CCV): A CCV must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.

10.9. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable. Refer to Section 10.13 for additional information.

10.10. Continuing Calibration Blank (CCB): The CCB consists of reagent water that is prepared per Section 11. A CCB must be analyzed after each CCV. If the CCB result is above the reporting limit, the CCB may be reanalyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable. Refer to Section 10.13 for additional information.

10.11. Reporting Limit Verification Standard (RLVS): The RLVS is a check standard at or below the concentration of the reporting limit. The RLVS must be analyzed at the beginning of an analytical run, after every 20 samples, and at the end of the analytical run. The acceptable recovery range for the RLVS is 50-150%.

10.12. RLVS Corrective Action: If an RLVS fails the acceptance criteria, another RLVS may be analyzed. If the second RLVS fails, then a new initial calibration curve must be analyzed. Sample associated with a failed RLVS must be reanalyzed. **Exception:** If the RLVS is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable. Refer to Section 10.13 for additional information.

10.13. Failure of the initial calibration, ICV, CCV, ICB or CCB that is due to improper or inadequate preparation requires the re-digestion and reanalysis of the associated preparation batch(es). Failure of the initial calibration, ICV, CCV, ICB or CCB due to instrument malfunction requires the instrument to be restored to proper working order and the reanalysis of samples associated with the failed QC.

11. Procedures

11.1. Aqueous Sample Preparation

11.1.1 Transfer a 30mL aliquot of well-mixed sample to a 50mL graduated digestion cup. Prepare an MS and MSD set by transferring 30mL aliquots of well-mixed sample to separate digestion cups and adding 1.5mL of the Intermediate #2 Mercury ICV/Spiking Standard (100ug/L) for a spike concentration of 5.0ug/L.

11.1.2 Prepare a Method Blank by adding 30mL of reagent water to a digestion cup. Prepare an LCS by adding 1.5mL of the Intermediate #2 Mercury ICV/Spiking Standard (100ug/L) to a digestion cup and diluting to 30mL with reagent water for a spike concentration of 5.0ug/L.

11.1.3 Add 0.75mL concentrated nitric to each digestion cup then add 1.5mL concentrated sulfuric acid acid to each digestion cup, mixing after each addition.

11.1.4 Add 5mL potassium permanganate solution to each digestion cup. Ensure that equal amounts of permanganate solution are added to Method Blank and LCS. Swirl to mix and add additional portions of permanganate solution, up to 15mL if necessary, until the purple color persists for at least 15 minutes. If the purple color does not persist after addition of 15mL of solution, then start over at Section 11.1.1 using a diluted aliquot of sample.

11.1.5 Add 2.5mL potassium persulfate solution to each digestion cup, cap loosely and heat samples for 2 hours in the Hot Block at 95°C.

11.1.6 Cool samples and add 1.8mL of the sodium chloride/hydroxylamine hydrochloride solution to each sample to reduce the excess potassium permanganate. **CAUTION**: perform this addition in a fume hood, as chlorine gas could be produced. Proceed to Section 11.4.

11.2. Solid Sample Preparation

11.2.1 Weigh 0.3g of sample into a 50mL digestion cup. To ensure the sample is representative of the entire container, the analyst should weigh out three 0.1g aliquots from different parts of the same container. Prepare an MS and MSD by weighing 0.3g portions of a sample into separate digestion cups and adding 1.5mL of the Intermediate #2 Mercury ICV/Spiking Standard (100ug/L) for a spike concentration of 0.5mg/Kg.

11.2.2 Prepare a Method Blank by placing several boiling chips in a digestion cup. Prepare an LCS by placing several boiling chips in a digestion cup and adding 1.5mL of the Intermediate #2 Mercury ICV/Spiking Standard (100ug/L) for a final concentration of 0.5mg/Kg.

11.2.3 Add 5mL of reagent water to each digestion cup.

11.2.4 Add 2.5mL of aqua regia to each digestion cup.

11.2.5 Heat samples for 2 minutes in the Hot Block at 95°C.

11.2.6 Cool samples and add 25mL reagent water then add 7.5mL potassium permanganate solution. Loosely cap each digestion cup.

11.2.7 Return the samples to the Hot Block and heat for 30 minutes at 95°C.

11.2.8 Cool samples again and add 3mL of the sodium chloride/hydroxylamine hydrochloride solution to each sample to reduce the excess potassium permanganate. **CAUTION**: perform this addition in a fume hood, as chlorine gas could be produced.

11.2.9 Adjust the digestate volumes to 50mL with reagent water and mix. Proceed to Section 11.4.

11.3. Calibration Standard Preparation

11.3.1 Prepare calibration standards in 50mL digestion cups per the instructions in Section 9.2.3.2.

11.3.2 Follow steps 11.1.3 through 11.1.6 to prepare calibration standards for aqueous matrix.

11.3.3 Follow steps 11.2.4 through 11.2.8 to prepare calibration standards for solid matrix.

11.4. Determination of Mercury

11.4.1 Configure the mercury analyzer according to manufacturer's instructions. Allow the colorimeter and recorder to warm up. Run a baseline with all reagents, using reagent water to flush the tubing. Whenever new tubing is used, allow ample time to flush the tubing.

11.4.2 Approximately 10mL portions of each standard, Method Blank, LCS, sample and MS/MSD are poured into autosampler tubes for analysis.

11.4.3 Establish initial calibration as described in Sections 10.1 through 10.5.

11.4.4 Once initial calibration is established, analyze each sample, Method Blank, LCS and MS/MSD. An example sequence may be as follows:

Initial calibration standards
ICV
ICB
RLVS
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB
RLVS

11.5 Calculations are performed directly by the instrument software. If dilutions were performed, the appropriate factors must be applied.

11.6 The instrument software calculates the amount of Mercury in the sample aliquot as follows:

$$X_s = (y - b)/a$$

Where: X_s = Concentration of the analyte
y = Total area or response of the analyte
a = slope of the line (the coefficient of x)
b = intercept of the line

11.7 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (mg/L)} = \frac{(X_s)(V_f)(D)}{(V_i)} \qquad \text{Solid Sample (mg/Kg)} = \frac{(X_s)(V_t)(D)}{(W_s)}$$

Where: X_s = Mercury concentration
V_f = Final sample volume in milliliters
D = Dilution factor
V_i = Initial sample volume in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

11.8 Any sample with a mercury concentration that exceeds the linear range of the calibration curve must be diluted with 3% HCl solution and re-analyzed or over range results must be qualified as estimated.

12 Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or boiling chips	One per preparation batch of up to 20 samples, per matrix.	Target analyte must be less than reporting limits	Re-digest and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analyte	One per preparation batch of up to 20 samples, per matrix.	80-120% Recovery	Re-digest and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analyte	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	75-125% Recovery ≤20% RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.

12.2 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13 Method Performance

13.1 **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually for each matrix per instrument.

13.2 **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14 Method Modifications

14.1 Digestion procedure modified to use digestion cups in a hot block instead of BOD bottles in a water bath.

14.2 Standards and some reagents purchased as certified solutions.

14.3 Stannous Chloride solution not stirred continually because it is a solution and not a suspension.

15 Pollution Prevention and Waste Management

15.1 Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

15.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 7470A and 7471A .

16.2 Pace Analytical Quality Manual; latest revision.

16.3 TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1 Not Applicable

18 Revisions

Document Number	Reason for Change	Date
IN-I-040-rev.9	1. Restructured document format to new corporate template. 2. Changed several sections of information to tabular form. 3. Section 9.1: revised vendor for potassium permanganate solution. 4. Section 9.2.3: intermediate standards are good for 6 months. 5. Section 9.2.3.2: revised working standard preparation directions. 6. Section 9.2.3.4: revised ICV preparation directions. 7. Added section 10.6 (low level check standard). 8. Table 12.1: revised language about frequency of blanks.	31Jan2007
S-IN-I-040-rev.10	1. Added new section 10.2 regarding how and when to drop points from an initial calibration curve. 2. Section 10.4: added last line regarding rerunning of samples. 3. Section 10.5: added "preceding or". 4. General: revised SOP references.	15Nov2007
S-IN-I-040-rev.11	1. Cover page converted to periodic review format. 2. Table of Contents fixed to include sections 10 and 11. 3. Section 3: added language that limits, volumes, vendors, are subject to change. 4. Table 7.1: updated to clarify hold time. 5. Section 8: updated and added "or equivalent" where applicable. 6. Section 9: updated and clarified separate standards as intermediate and working. Created a table for working calibration standard examples. 7. Section 10: completely revised to individually clarify calibration requirement including corrective actions. 8. Section 11: clarified procedure including intended run sequence and added calculations for final concentration. 9. Section 12: updated limits and corrective actions. 10. Section 15: changed NELAC to TNI.	19Jun2011
S-IN-I-040-rev.12	1. Section 3.2: added reference to MDLs and revised RL for solids. 2. Section 9.1: added potassium persulfate, wash solution and boiling chips. Revised or expanded other reagents. 3. Table 9.2: added RLVS 4. Table 9.3: added RLVS 5. Section 9.2.3.2: added RLVS 6. Section 10: added RLVS 7. Section 11.1.5: added that digestion cups be capped loosely for digestion. 8. Section 11.4.4: added RLVS 9. Section 11: added that calculations are performed by instrument software. 10. Section 11.8: added that over range results must be qualified. 11. Table 12.1: revised method blank corrective action. 12. Inserted new Method Modifications section.	27Sep2012
S-IN-I-040-rev.13	1. Table 9.1: revised details of Stannous Chloride reagent use and handling. 2. Section 14: added a modification for no continuous stirring of Stannous Chloride.	29Oct2012

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STANDARD OPERATING PROCEDURE

TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)
REFERENCE METHOD: EPA SW-846 METHOD 1311

SOP NUMBER:	S-IN-I-062-rev.10
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-I-062-rev.9

APPROVAL

General Manager — October 18, 2012 Date

Quality Manager — October 18, 2012 Date

Department Manager — October 18, 2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for extracting organic and inorganic compounds from samples using the Toxicity Characteristic Leaching Procedure (TCLP) as stated in EPA SW-846 Method 1311.

2. Summary of Method

2.1. An aliquot of waste sample is tumbled in a sealed vessel containing extraction fluid to leach out compounds of interest from the sample. The leachate is then the sample that is analyzed by the required determinative method.

3. Scope and Application

3.1. The TCLP is designed to determine the mobility of organic and inorganic constituents present in liquid, solid and multiphasic wastes.

3.2. If a total analysis of the waste sample demonstrates that individual analytes are not present or are at such low levels that the regulatory limits will not be exceeded, the TCLP need not be run.

3.3. If the analysis of any liquid fraction of the waste shows analytes of concern at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory limit for that compound, then the remaining fractions do not need to be analyzed.

3.4. Bottle extractors are used for non-volatile analytes and a special ZHE extraction device is used to facilitate extraction of samples for volatile analysis.

3.5. Extraction using the Zero Headspace Extractor (ZHE) for volatiles is not necessary if an analysis of the bottle extract shows a concentration of a regulated volatile compound above the regulatory limit for a compound. The bottle extract cannot be used to demonstrate that the concentrations of volatile compounds are below regulatory limits.

3.6. Liquid wastes, those containing <0.5% dry solid material, after filtration through a 0.6 to 0.8um glass filter, are defined as the TCLP extract.

3.7. For wastes containing $\geq$0.5% solids, the liquid portion, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of the appropriate extraction fluid equal to 20 times the weight of the solid phase.

3.8. If compatible, the initial liquid phase and the extracted water from the solid phase are combined and analyzed as one sample. If the wastes are not compatible, they are analyzed separately and results are mathematically combined to give a volume-weighted average concentration.

3.9. Vendors, equipment and supplies are subject to change.

3.10. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of TCLP extraction equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. No significant interferences are listed for the preparation of the TCLP extracts. Potential interferences during analyses are discussed in the individual determinative method SOP's.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The TCLP reagents are toxic and should be handled with extreme care.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Solids and Semi-solids	Non-volatiles: >200g preferably in a glass container (to avoid phthalates in SVOA analysis) Volatiles: >50g in a glass container	No preservation required	0° to 6°C Unless refrigeration causes irreversible physical change.	**VOA:** extract for TCLP within 14 days of collection date and analyze for VOA within 14 days of TCLP extraction date. **SVOA:** extract for TCLP within 14 days of collection date, extract for SVOA within 7 days of TCLP extraction date and analyze for SVOA within 40 days of SVOA extraction date. **Mercury:** extract for TCLP within 28 days of collection date and analyze for Hg within 28 days of TCLP extraction date. **Metals:** extract for TCLP within 180 days of collection date and analyze for metals within 180 days of TCLP extraction date.

Samples should be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment and Supplies

Equipment	Description / Comments
Zero Headspace Extraction (ZHE) vessels	Used for volatile extractions; must have an internal volume of 500-600mL of sample and be equipped to accommodate a 90-110mm filter. ZHEs contain o-rings that should be replaced frequently. The piston within the ZHE should be able to be moved with approx. 15psi or less. Refer to Attachment 1.
Bottle Extraction vessels	Used for non-volatile extractions; borosilicate glass bottles of sufficient size to fit into the agitation apparatus. Plastic bottles shall not be used if organics are to be determined.
Agitation apparatus	Device capable of rotating the extraction vessels in an end-over-end fashion at 30 +/- 2rpm. Refer to Attachment 1.
Filtration Device	For non-volatile extractions; the filtration device should have a minimum internal volume of 300mL, be able to accommodate a minimum filter size of 47mm, must be capable of exerting pressures of up to 50psi or more and shall be constructed of inert material.
Filters	Filters shall be made of borosilicate glass fiber, shall contain no binder material, and shall have an effective pore size of 0.6 to 0.8um, or equivalent.
pH meter	Accurate to +/- 0.05 units at 25^{o}C
Gas-tight syringes	Used for ZHE extract transfer
Glass VOA vials	40mL; used for ZHE extract collection
Tedlar bags	Used for ZHE extract collection
Analytical balance	Accurate to within +/- 0.01g
Glass beakers	250mL or equivalent
Watch glasses	Appropriate diameter to cover the beaker
Magnetic stirrer	Including stir bars

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
Hydrochloric acid (1N)	Reagent grade
Nitric acid (1N)	Reagent grade
Sodium Hydroxide (1N)	Reagent grade
Glacial Acetic acid	Reagent grade
Extraction Fluid #1	Add 5.7mL of glacial acetic acid to 500mL of reagent water then add 64.3mL of 1N sodium hydroxide and dilute to 1L with reagent water. **When correctly prepared, the pH of this fluid will be 4.93 +/- 0.05. The pH should be checked prior to use. If the pH is not 4.93 +/-0.05, the fluid shall be discarded and fresh extraction fluid prepared.**
Extraction Fluid #2	Dilute 5.7mL of glacial acetic acid to 1L with reagent water. **When correctly prepared, the pH of this fluid will be 2.88 +/- 0.05. The pH should be checked prior to use. If the pH is not 2.88 +/- 0.05, the fluid shall be discarded and fresh extraction fluid prepared.**

9.2. Analytical Standards

9.2.1. Not applicable to this SOP.

10. Calibration

10.1. pH meter must be calibrated according to applicable SOP or manufacturer's instructions.

11. Procedures

11.1. Preliminary Evaluations

Preliminary evaluations must be made on a 100g aliquot of the waste sample. If 100g is not available, client consent is required to perform the extraction using less than 100g. These evaluations include determination of the percent solids of the sample; determination of whether the waste sample contains insignificant solids and is therefore its own extract after filtration; determination if there is a need for particle size reduction; and determination of which of the two extraction fluids are required. Refer to Attachment 2 for additional information.

11.1.1 Percent Solids Determination:

11.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration, indicating that the waste is 100% solids, then skip to Section 11.1.2.

11.1.1.2 If the sample is a liquid or is multiphasic, separation to make a preliminary determination of percent solids is required. This involves filtration as follows:

11.1.1.2.1 Pre-weigh the filter to be used and the container that will collect the filtrate.

11.1.1.2.2 Assemble the filtering device per manufacturer's specifications and place the filter on the support screen.

11.1.1.2.3 Weigh out a subsample of the waste, 100g minimum, record the exact weight.

11.1.1.2.4 Allow slurries to stand to permit the solid phase to settle.

11.1.1.2.5 Quantitatively transfer the sample to the filtration device. Begin applying gentle vacuum or pressure to the filtration device of 1-10psi until air or pressurized gas moves through the filter. If air does not pass through the filter or if no additional liquid comes through in a 2 minute period, slowly increase the pressure in 10psi increments to a maximum of 50psi until liquid flow has ceased. The material in the filter holder is defined as the solid phase of the waste and the filtrate is defined as the liquid phase. Use only one filter per sample.

11.1.1.2.6 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container. Determine the weight of the solid phase by subtracting the weight of the pre-weighed filter from the total weight of the filter with filtered solids. Record the weight of the liquid and solid phases and calculate the percent solids as follows:

$$\textbf{Percent solids} = \frac{\textbf{weight of solid phase}}{\textbf{total weight of sample}} \times 100$$

11.1.1.2.7 If the percent solids determined in Section 11.1.1.2.6 is $\geq$0.5%, then proceed either to Section 11.1.2 to determine if the material requires particle size reduction or to Section 11.1.1.2.8 if it is

noticed that an amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 11.1.1.2.6 is <0.5%, then proceed to Section 11.2.2.1 if non-volatiles TCLP is to be performed and to Section 11.3.5 with a fresh portion of the waste if volatile TCLP is to be performed.

11.1.1.2.8 If it appears that the filtrate is entrained in wetting of the filter, remove the solid phase and filter from the filtration apparatus and dry the filter and solid phase at 100 +/-20°C until two successive weighings yield the same value within +/-1%. Record the final weight. Calculate the percent dry solids as follows:

$$\textbf{Percent dry solids} = \frac{\textbf{(weight of dry waste + filter) – tared wt. of filter}}{\textbf{Initial weight of waste}} \times 100$$

11.1.1.2.9 If the percent dry solids determined is <0.5%, proceed to Section 11.2.2.1if non-volatile TCLP is to be performed, and to Section 11.3.5 if volatile TCLP is to be performed. If the percent dry solids is ≥0.5%, and if non-volatile TCLP is to be performed, proceed to the Section 11.1.2 with a fresh portion of waste. If only volatile TCLP is to be performed, proceed to Section 11.3.5.

11.1.2 Particle Size Reduction Determination: this is required unless the solid portion of the sample is capable of passing through a minimum 9.5mm sieve. If it will not pass through, the sample must be ground, crushed or cut until it will pass through.

11.1.3 Extraction Fluid Determination: For determining pH, refer to SOP S-IN-I-024 or its replacement. For inorganic analysis, the following steps are used to determine the appropriate fluid:

11.1.3.1 If the sample was found to have greater >0.5 % solids, weigh out a 5g aliquot of the solid phase of the waste into a 250mL beaker.

11.1.3.2 Add 96.5mL of reagent water to the beaker, cover with a watchglass, and stir for 5 minutes using a magnetic stirrer. Determine and record the pH. If the pH is <5.0, use extraction fluid #1.

11.1.3.3 If the pH is >5.0, add 3.5mL of 1N HCl, stir briefly and cover with a watchglass. Heat without stirring to 50°C and hold at 50°C for 10 minutes.

11.1.3.4 Let the solution cool to room temperature and determine the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

11.2 Extraction Procedure for Non-volatile Analysis

A minimum sample size of 100g is recommended. In some cases, a larger sample size may be appropriate, depending on solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics and organics are analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction is insufficient, additional extractions may be performed and the extracts from each combined and aliquoted for analysis. If a minimum of 100g of sample is not available, the client must be contacted. Before the extraction can proceed, the client must return a signed copy of the *TCLP Deficiency Release,* as seen in Attachment 3. Refer to Attachment 2 for additional information.

11.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration, indicating that the waste is 100% solids, transfer a minimum 100g aliquot of the waste directly into an extractor bottle and proceed to Section 11.2.3.

11.2.2 If the sample is liquid or multiphasic, separation is required per the filtration procedure described in Section 11.1.1.2.

11.2.2.1 Transfer the solid material resulting from the filtration procedure, including the filter, into the bottle extractor and proceed to Section 11.2.3. Refer to Section 11.2.7 regarding handling of the filtrate.

11.2.3 Determine the amount of extraction fluid to add to the extraction vessel as follows:

$$\textbf{Weight of extraction fluid} = \frac{\textbf{20 x percent solids x unfiltered sample weight}}{\textbf{100}}$$

11.2.4 Slowly add this amount of the appropriate extraction fluid to the extractor bottle. Close the bottle tightly with a lid. Secure the sealed extractor bottle in the agitation device and rotate at 30 +/- 2rpm for 18 +/-2 hours. The temperature of the room where TCLP extractions are performed shall be maintained at 23 +/-2^{o}C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle. To relieve excess pressure, the extractor bottle may be periodically opened and vented into a hood.

11.2.5 Following the 16-20 hour tumble, the liquid portion of the extract is separated from the solid material by filtration, using a clean glass filter. More than one filter may be used to facilitate final filtering of the TCLP extract, if necessary. Filters shall be acid rinsed with 1N nitric acid if metals are to be determined, or use an acid pre-washed filter from supplier.

11.2.6 If the original waste contained no liquid, then the tumbled extract is defined as the TCLP extract.

11.2.7 If compatible, combine the liquid extract resulting from Section 11.2.5 with the initial liquid phase of the waste obtained in Section 11.2.2.1. This combined liquid is then defined as the TCLP extract. If the two liquid fractions are not compatible, analyze the two fractions separately and combine their results mathematically.

11.2.8 Following collection of the TCLP extract, the pH of the extract should be determined and recorded. Immediately aliquot the extract for metals analysis, if applicable, and preserve with HNO_3 to a pH of <2. **Note**: An aliquot of sample to be spiked for metals should be spiked before preserving with nitric acid.

11.2.9 All other aliquots must be stored under refrigeration until analyzed. The TCLP extract shall be prepared and analyzed according to the appropriate analytical method.

11.2.10 TCLP extracts to be analyzed for metals must undergo acid digestion, unless the analysis of the undigested aliquot produces results for metallic analytes above the regulatory limits. Data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous.

11.2.11 If the individual phases are to be analyzed separately, determine the volume of the individual phases (to +/-0.5%), conduct the appropriate analysis, and combine the results mathematically by using a simple volume-weighted average:

$$\textbf{Final analyte concentration} = \frac{\textbf{(V1) (C1) + (V2) (C2)}}{\textbf{V1 + V2}}$$

Where: V1 = The volume of the first phase, L
C1 = The concentration of the analyte of concern in the first phase, mg/L
V2 = The volume of the second phase, L
C2 = The concentration of the analyte of concern in the second phase, mg/L

11.3 Extraction Procedure for Volatile Analysis

Use the ZHE device to obtain the TCLP extract for analysis of volatile compounds. The ZHE device has approximately a 500mL internal capacity and can accommodate a maximum of 25g of solid. Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Repeated filling of the ZHE to obtain 25g of solid is not permitted. Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold to minimize loss of volatiles. All samples for TCLP Volatiles analysis must be prepared by means of extraction and/or filtration through the ZHE apparatus. Refer to Attachment 2 for additional information.

11.3.1 Pre-weigh the filtrate collection container and set aside.

11.3.2 Before use each ZHE extractor must be pressure tested. Fully assemble each ZHE extractor without the piston and charge to 50psi. Maintain the ZHE at 50psi for 15 minutes in order to verify its suitability for use. Section 4.2.1 of Method 1311 indicates that the ZHE must hold 50psi for 1 hour?

11.3.3 Place the ZHE piston within the body of the ZHE to a height that minimizes the distance the piston must move once the ZHE is charged with sample. Secure the glass fiber filter between the support screens and set aside.

11.3.4 If the waste is 100% solids, weigh out a subsample of a maximum of 25g. Record the weight and proceed to Section 11.3.6.

11.3.5 If the waste contains <0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing ≥0.5% dry solids, use the percent solids information obtained in Section 11.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

11.3.5.1 For wastes containing <5% solids, weigh out a 500g subsample of waste and record the weight.

11.3.5.2 For wastes containing ≥5% solids, determine the amount of waste to charge into the ZHE as follows:

$$\textbf{Weight of waste to charge ZHE} = \frac{25}{\text{Percent Solids}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

11.3.6 Perform particle size reduction per Section 11.1.2, if necessary. If reduction of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible. Sieving of the waste is not recommended due to the possibility that volatiles may be lost.

11.3.7 Quantitatively transfer the entire sample, liquid and solid phases, quickly to the ZHE. Secure the filter and support screens then secure the top flange. Tighten all ZHE fittings.

11.3.8 Attach the gas line to the gas inlet/outlet valve on the bottom flange making sure the liquid inlet/outlet valve on the top flange is open. Begin applying gentle pressure of 1-10psi (or more if necessary to a maximum of 50psi) to force all headspace slowly out of the ZHE. This operation should be performed

in a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure.

11.3.9 If the waste sample in the ZHE contains a liquid phase, attach an evacuated, pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10psi to force the liquid phase of the sample into the filtrate collection container. It no additional liquid has passed through the filter in any 2 minute period, slowly increase the pressure in 10psi increments to a maximum of 50 psi until liquid flow has ceased. Close the liquid inlet/outlet valve, discontinue pressure to the piston and disconnect and weigh the filtrate collection container.

11.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate, if any, is defined as the liquid phase. If the original waste contained <0.5% dry solids, the filtrate is defined as the TCLP extract and is analyzed directly. The liquid phase may now be either analyzed immediately or refrigerated under minimal headspace conditions until the time of analysis.

11.3.11 Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\textbf{Weight of extraction fluid \#1} = \frac{\textbf{20 x percent solids x weight of waste filtered}}{\textbf{100}}$$

11.3.12 With the ZHE in the vertical position, release gas pressure on the ZHE piston and deliver the appropriate amount of extraction fluid #1 into the ZHE through the liquid inlet/outlet valve. Once the extraction fluid has been added, immediately close the liquid inlet/outlet valve. Check the ZHE to ensure that all valves are closed. Manually rotate the ZHE in an end-over-end fashion 2 or 3 times. With the ZHE in the vertical position, pressurize the ZHE to 5-10psi and slowly open the liquid inlet/outlet valve to bleed off any headspace. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed. Record the final pressure.

11.3.13 Place the ZHE in the agitation apparatus and rotate the ZHE for 18 +/-2 hours at 30 +/-2 rpm. Temperature of the room where tumbling occurs shall be maintained at 23 +/-2^{o}C. Record the range of temperatures during the entire tumbling period.

11.3.14 Following the 16-20 hour agitation period, check the pressure in the ZHE by quickly opening an closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained and no gas release is noted, perform the extraction again with a new sample of waste. Filter the extract through the glass fiber filter using the ZHE device as discussed in Section 11.3.9. All extract should be filtered and collected if the extract is multiphasic or if the waste contained an initial liquid phase. If the original waste contained no initial liquid phase, the filtrate is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtrate and the initial liquid phase are collectively defined as the TCLP extract.

11.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store refrigerated with minimal headspace. If the individual phases are to be analyzed separately, determine the volume of the individual phases, conduct the analysis and combine the results mathematically using the equation shown in Section 11.2.1.

12 Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Extraction (Tumble) Blank	Extraction fluid	VOA: Minimum of 1 blank per 20 extractions per extraction vessel unless client requests a tumble blank per batch. SVOA: blank is tumbled per batch of extraction fluid unless client requests a tumble blank per batch.	Target analytes must be less than reporting limits	Re-tumble associated samples (if possible) if blank result is greater than RL. If not, blank and samples are reported with qualifiers. If blank result is >RL but associated sample(s) are ND, no action is required.
Method Blanks (MB)	See determinative method (extraction blank filtrate should be analyzed per each analytical method)	See determinative method	See determinative method	See determinative method
Laboratory Control Sample (LCS)	See determinative method (extraction blank filtrate should be used per analytical method)	See determinative method	See determinative method	See determinative method
Matrix Spike (MS)	See determinative method	A minimum of one MS must be analyzed per batch per waste stream. Matrix spike solution is added after filtration but before any preservation of sample.	See determinative method	See determinative method

12.2 The required rotation speed of the agitation device is 30 +/-2rpm. The rotation speed is checked monthly and recorded on a monitoring sheet. A supervisor should be alerted if the rotation speed is unacceptable and corrective actions must be taken.

13 Method Performance

13.1 The TCLP analysts must read and understand this procedure with written documentation maintained in their training files.

14 Method Modifications

14.1 Samples are not particle sized to 1mm prior to extraction fluid determination.

15 Pollution Prevention and Waste Management

15.1 Procedures for handling waste generated during this analysis are addressed in ALL-S-002, Waste Handling or other applicable SOP.

15.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

16.1 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Method 1311, Toxicity Characteristic Leaching Procedure; 3rd edition; December 1996.

16.2 Pace Analytical Quality Manual; latest revision.

16.3 TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1 Attachment 1:Diagram of agitation apparatus and ZHE apparatus

17.2 Attachment 2: TCLP Preliminary Evaluations Flow Diagram

17.3 Attachment 3: TCLP Deficiency Release Form

18 Revisions

Document Number	Reason for Change	Date
S-IN-I-062-rev.6	1. Restructured document format to new corporate template. 2. Changed several sections of information to tabular form. 3. Sections 11.6 and 11.16: added language about minimum/maximum thermometer now in use. 4. Section 13: removed references for MDL and DOC (not applicable to the TCLP tumbling method). 5. Table 9.1: revised catalog numbers.	21Aug2007
S-IN-I-062-rev.8	1. Section 8: sections 8.1 and 8.2 combined into a single table. 2. Section 9.1: "or equivalent" added to specific reagent catalog numbers. 3. Section 11.4: expanded description of pH determination procedure. 4. Section 11.16: removed reference to min/max thermometer. 5. Section 16.2: removed	06May2009
S-IN-I-062-rev.9	1. Cover sheet changed to periodic review format. 2. Section 3.6: >0.5% changed to ≥0.5% 3. Table 7.1: sample amounts increased, hold times clarified, storage changed from 4 +/-2°C to 0° to 6°C. 4. Table 8.1: expanded descriptions and added "or equivalent" where applicable. 5. Table 9.1: removed vendor column and expanded descriptions. Included extraction fluid requirements. 6. Section 11: separated into sections for preliminary evaluations, non-volatile extraction and volatile extraction, expanded to include step-by-step instructions with references to Attachment 2, included equations and added requirements that were not included in previous versions. 7. Table 12.1: removed MSD 8. Section 12.3: removed and included this information in Section 11. 9. Section 16: added two attachments.	05Aug2010

S-IN-I-062-rev.9	1. Section 3: added language that vendors, equipment, supplies, etc…are subject to change. 2. Section 10: added that pH meter must be calibrated. 3. Section 11.1.3: added reference to SOP for pH procedure. 4. Table 12.1: revised extraction blank corrective action and clarified that method blank and LCS are to be prepared using the extraction blank filtrate. 5. Inserted new Method Modifications section 6. References: changed NELAC to TNI	18Oct2012

Attachment 1

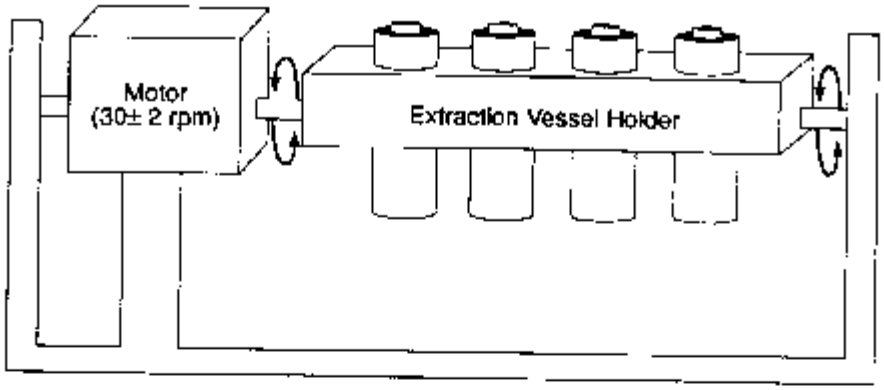


Figure 1. Rotary Agitation Apparatus

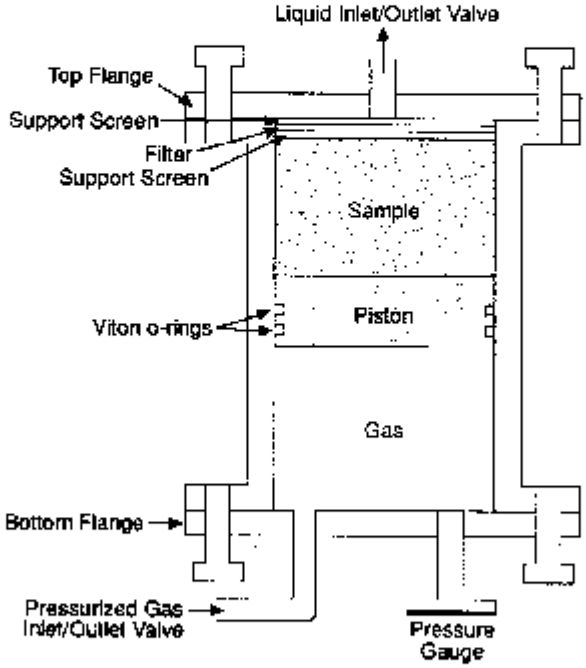
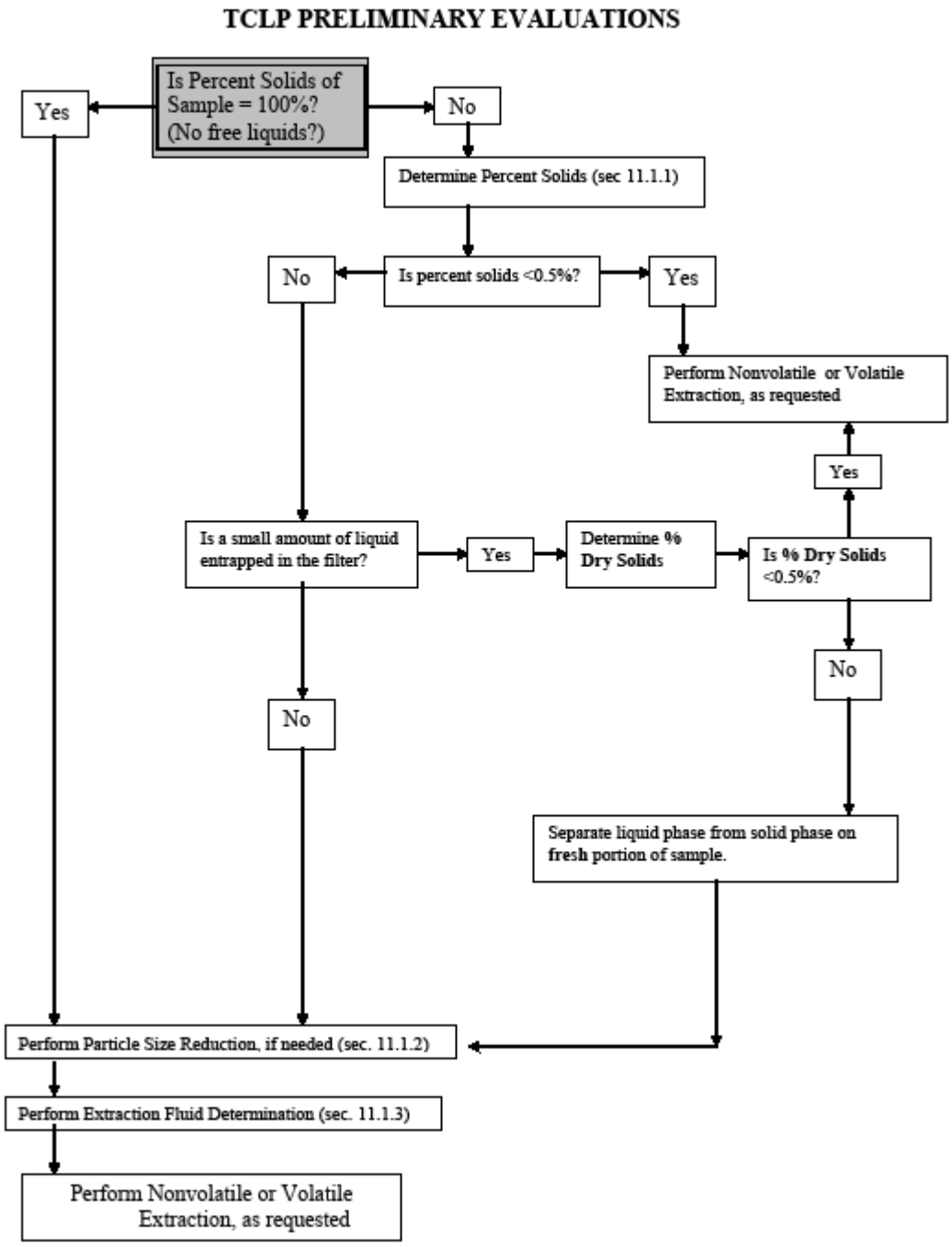


Figure 2. Zero-Headspace Extractor (ZHE)

Attachment 2



Attachment 3

Pace Analytical Services, Inc.
7726 Moller Road
Indianapolis, IN 46268
Phone: 317-875-5894
Fax: 317-872-6189

TCLP DEFICIENCY RELEASE

DATE:

TO:

RE: NOTE OF TCLP DEFICIENCY

This memo is in notice of a deficiency in the TCLP extraction for the sample identified:

PROJECT# **LAB#:**

CLIENT ID:

In accordance with the TCLP method, the required weight to be used with a sample is 100 grams. The sample identified above does not have sufficient weight to comply with this requirement. In order to proceed with the TCLP extraction, your written approval is needed. The sample will then be processed with a reduced weight and the extraction fluid volume adjusted accordingly. The analytical report will indicate the deviation with a qualifier for the sample.

Should your decision be to provide additional sample for extraction, please contact your customer service representative to request containers, evaluate holding time constraints, or specify turnaround time needs.

Thank you.

Approval to proceed with TCLP extraction using reduced weight has been granted by:

Signature: _____

Name (Print): _____

Date: _____

F-IN-C293-rev.0, 05Aug2010

Pace Analytical Services, Inc.
7726 Moller Road
Indianapolis, IN 46268

Phone: 317.875.5894
Fax: 317.872.6189

STANDARD OPERATING PROCEDURE

THE DETERMINATION OF HEXAVALENT CHROMIUM

REFERENCE METHODS: EPA SW-846 METHOD 7196A AND STANDARD METHODS 3500-CR B

SOP NUMBER:	S-IN-I-063-rev.11
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-I-063-rev.10

APPROVAL

Karl Anderson
General Manager — September 27, 2012 Date

Bud Schrage
Quality Manager — September 26, 2012 Date

David D. Mark
Department Manager — September 26, 2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining Hexavalent Chromium in aqueous and solid samples while meeting the requirements specified in EPA SW-846 method 7196A and Standard Methods 3500-Cr B. **NOTE: Standard Methods 3500-Cr B supersedes and is equivalent to Standard Methods 3500-Cr D**.

2. Summary of Method

2.1. Dissolved hexavalent chromium is determined colorimetrically by reaction when diphenylcarbazide in an acid solution. A red-violet color is produced and is measured colorimetrically at 540nm.

3. Scope and Application

3.1. This method is applicable for the measurement of hexavalent chromium in groundwater, surface water, soil, sediment and domestic and industrial wastes. Refer to SOP S-IN-I-070 Alkaline Digestion of Solid Samples for Hexavalent Chromium or its replacement for procedures associated with the preparation of solid samples.

3.2. The reporting limit for Hexavalent Chromium is 0.01mg/L for aqueous samples and 4mg/kg for solid samples. Refer to the LIMS for method detection limits.

3.3. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of hexavalent chromium analysis equipment and reagents. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. The chromium reaction with the diphenylcarbazide is usually free from interferences. However, hexavalent molybdenum and mercury salts also react to form color complexes with the reagent. Vanadium can also interfere, but concentrations up to 10 times that of chromium can be tolerated.

5. Safety

5.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock hexavalent chromium standards are toxic and should be handled with extreme care.

5.2. **Samples**: Take precautions when handling samples. Samples must always be treated as potentially hazardous “unknowns”. The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All digestions must be conducted under a fume hood.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous Unpreserved	250mL in plastic container	None required	0°C to 6°C	Analyze samples within 24 hours of collection date.
Aqueous Preserved	250mL in plastic container	NaOH to pH >9 Alternatively, Ammonium Sulfate Buffer to pH >9	0°C to 6°C	Analyze samples within 28 days of collection date.
Solid	>100g in a glass container	None required	0°C to 6°C	Digest samples within 30 days of collection date and analyze digestates within 7 days of digestion date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Description / Comments
Spectrophotometer	Hach DR5000 or equivalent	For use at 540nm with a flow-through cell providing a light path of 1cm or longer

8.2. General Supplies

Item	Vendor	Description
Graduated Cylinder		Class A, 50mL
Mechanical pipets	Eppendorf or equivalent	Various sizes
Filter membrane	Whatman or equivalent	0.45um
Disposable beakers		20mL
Transfer pipets		Disposable

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II
ChromaVer 3 Chromium reagent powder pillows	Hach cat.#12710-99 or equivalent
1:1 Sulfuric Acid	Reagent grade or equivalent
Sodium Hydroxide (50%)	Fisher cat#SS254-4 or equivalent
Ammonium Sulfate Crystal	Fisher cat#A702-500 or equivalent
Ammonium Hydroxide	Fisher cat#A512-500 or equivalent
Ammonium Sulfate Buffer	Dissolve 33 g of ammonium sulfate in 75 mL reagent water and add 6.5 mL ammonium hydroxide. Dilute to 100 mL with reagent water.

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains all target analytes and should be prepared from a different source than the calibration standards.	This solution is used for the LCS, MS/MSD, and post-digestion spikes.

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Cr(VI) Calibration Standard	Ricca catalog #2095-47; 100mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate Cr(VI) Calibration Standard	Refer to Section 9.2.3.1	Must be prepared fresh daily	Not Applicable
Working Cr(VI) Calibration Standards	Refer to Section 9.2.3.2	Must be prepared fresh daily	Not Applicable
Stock Cr(VI) ICV/LCS Standard	Hach; catalog #810-42H; 50mg/L or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions

9.2.3. Standard Preparation Procedures

Refer to the standard preparation logbook or database for additional instructions regarding preparation of standards for Cr(VI) analysis. Instructions for preparation of fresh daily standards are detailed below. Refer to SOP S-IN-I-070 Alkaline Digestion of Solid Samples for Hexavalent Chromium or its replacement for reagents and standards associated with the preparation of solid samples.

9.2.3.1. Intermediate Cr(VI) Calibration Standard Preparation

Dilute 1mL of the Stock Cr(VI) Calibration Standard (100mg/L) to 100mL with reagent water to give a final concentration of 1.0mg/L.

9.2.3.2. Working Cr(VI) Calibration Standard Preparation

Working calibration standards must be prepared fresh daily by diluting the Intermediate Cr(VI) Calibration Standard (1mg/L) to 10mL with reagent water. Examples of possible calibration standards are as follows:

Standard ID	Amt. of Intermediate Calibration Std. Used	Final Volume	Final Concentration
Standard 1	0.1mL	10mL	0.01mg/L
Standard 2	0.5mL	10mL	0.05mg/L
Standard 3	1.0mL	10mL	0.10mg/L
Standard 4 (CCV)	5.0mL	10mL	0.50mg/L
Standard 5	7.5mL	10mL	0.75mg/L
Standard 6	10mL	No dilution required	1.0mg/L

9.2.3.3. Working Cr(VI) ICV/LCS Standard Preparation

Dilute 0.1mL of the Stock Cr(VI) ICV/LCS Standard (50mg/L) to 10mL with reagent water for an ICV concentration of 0.50mg/L. This standard must be prepared fresh daily. This standard is also used as the LCS.

10. Calibration

10.1. Initial Calibration: A minimum of 5 calibration standards is required. The lowest calibration standard must be at or below the reporting limit. A new initial calibration must be analyzed every 6 months at a minimum. Refer to the Quality Manual for more information regarding calibration curves.

10.2. Linear Calibration: After zeroing the spectrophotometer with a reagent blank, use the instrumentation software to prepare a standard curve by plotting absorbance versus Cr(VI) concentration of each calibration standard. The analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.3. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.995.

$$r = \frac{\sum_{i=1}^{N} (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} (X_i - \overline{X})^2 \right) * \left(\sum_{i=1}^{N} (Y_i - \overline{Y})^2 \right)}}$$

10.4. Initial Calibration Corrective Action: If the curve does not meet the acceptance criteria, then a new calibration curve must be analyzed. If the second curve attempt does not meet the acceptance criteria, the analyst must consult the department manager and instrument maintenance and/or preparation of new standards must be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.5. Initial Calibration Verification (ICV): In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve. Acceptable recovery range for the ICV is 90-110%.

10.6. ICV Corrective Action: If the ICV is not acceptable, another ICV may be analyzed. If the second ICV fails, then a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new standards must also be considered. Samples associated with a failed ICV must be reanalyzed. Exception: If the ICV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

10.7. Continuing Calibration Verification (CCV): When an ICAL is not analyzed, the calibration must be verified by analyzing a CCV at the beginning of the analytical sequence. In all cases, a CCV must also be analyzed after every 10 samples and at the end of the analytical sequence to verify the system is still calibrated. The CCV should be from the same material as the curve standards. The acceptable recovery range for the CCV is 90-110%.

10.8. CCV Corrective Action: If a CCV fails the acceptance criteria, another CCV may be analyzed. If the second CCV fails, then a new initial calibration curve must be analyzed. Samples must be bracketed by acceptable CCVs in order to be reportable. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL are reportable.

10.9. Continuing Calibration Blank (CCB): A CCB consists of 10mL reagent water to which a Chromaver 3 reagent powder pillow has been added. A CCB must be analyzed after each ICV or CCV. If the CCB result is above the reporting limit, another CCB may be analyzed. If the second CCB fails, then a new calibration curve must be analyzed. Samples associated with a failed CCB must be reanalyzed. **Exception**: If the CCB is >RL, associated samples determined to be <RL are reportable.

11. Procedures

11.1. If samples are turbid, centrifuge samples to remove turbidity. If after centrifugation samples remain turbid, filtration of samples through a 0.45um filter may be performed.

11.2. Unpreserved aqueous samples: Place 10mL of sample into a 20mL disposable beaker and add contents of one Chromaver 3 chromium reagent powder pillow. Cap the container and shake or stir for about 30 seconds to dissolve the powder.

11.3. Preserved aqueous samples and soil digestates: Place 10mL of aqueous sample or soil digestate into a 20mL disposable beaker. Add 0.2mL of 1:1 sulfuric acid and add the contents of one Chromaver 3 chromium reagent powder pillow. Cap the container and shake or stir for about 30 seconds to dissolve the powder.

11.4. Allow 5 to 10 minutes for the color to develop but do not wait beyond 20 minutes to take the reading of the sample on the spectrophotometer. A red-violet color will be observed in the presence of Cr(VI).

11.5. Adjust the wavelength control of the spectrophotometer to 540nm. Zero the spectrophotometer using the reagent blank. Measure the absorbance of the standards, samples and blanks. A typical run sequence may be as follows:

ICAL Standards
ICV
(If ICAL not run, CCV would replace the ICAL and the ICV in the sequence)
CCB
Method blank
LCS
Client samples
CCV
CCB
Client samples
CCV
CCB

11.6. To correct for background in samples, use an aliquot of the sample containing all reagents except the Chromaver 3 reagent powder pillow.

11.7. Any sample with a Cr(VI) concentration that exceeds the linear range of the calibration curve must be diluted and reanalyzed or over range results must be qualified as estimated.

11.8. From the corrected absorbance, determine the concentration of chromium present using the calculation below:

$$X = [(y)(a)] + b$$

Where: X = sample concentration
y = response or absorbance, corrected for background
a = slope
b = y-intercept

11.9. Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (mg/L)} = \frac{(X)(V_f)(D)}{(V_i)} \qquad \text{Solid Sample (mg/Kg)} = \frac{(X)(V_t)(D)}{(W_s)}$$

Where: X = Sample concentration
V_f = Final sample volume in milliliters
D = Dilution factor
V_i = Initial sample volume in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent or Ottawa sand	One per preparation batch of up to 20 samples per matrix.	Target analytes must be less than reporting limits	Re-digest and reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Cr(VI)	One per preparation batch of up to 20 samples per matrix.	80-120% Recovery	Re-digest and reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Aqueous Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Cr(VI)	One MS/MSD set per batch of up to 20 samples for waters.	85-115% Recovery ≤20%RPD	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Solid Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Cr(VI)	One **soluble** and one **insoluble** MS/MSD set per preparation batch of up to 20 samples for solids.	75-125% Recovery ≤20%RPD	If outside acceptance limits, evaluate sample for pH and Eh and compare to Figure 2 in Method 3060A. If in reducing range, no re-digestion is required. If outside reducing range, re-digestion and reanalysis of entire preparation batch is required. Refer to SOP S-IN-I-069 or equivalent for pH procedure. Refer to SOP S-IN-I-035 or equivalent for Eh procedure.
Sample Duplicate (DUP) **Solids only**	Sample	One Sample Duplicate per preparation batch of up to 20 samples.	≤20% RPD	No corrective action is necessary if sample concentration is <4x RL. If sample concentration is >4x RL, the RPD must be qualified appropriately.
Post-Digestion Matrix Spike **Solids only**	Cr(VI) at 40mg/Kg or 2x sample conc.	One per preparation batch of up to 20 samples.	85-115% Recovery	No corrective actions necessary.

12.2. Aqueous Method Blank Preparation: the Method Blank consists 10mL of reagent water.

12.3. Aqueous LCS Preparation: Dilute 0.1mL of the Stock Cr(VI) ICV/LCS Standard (50mg/L) to 10mL with reagent water for an LCS concentration of 0.50mg/L. Prepare fresh daily.

12.4. Aqueous MS/MSD Preparation: Dilute 0.1mL of the Stock Cr(VI) ICV/LCS Standard (50mg/L) to 10mL with sample for a spike concentration of 0.50mg/L.

12.5. LCS equation:

$$\mathbf{R = (C/S) * 100}$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.6. MS/MSD equation:

$$\mathbf{R = \frac{(Cs - C)}{S} * 100}$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$\mathbf{RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100}$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13. Method Performance

13.1. Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

13.2. Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14. Method Modifications

14.1. Aqueous sample preservation to pH >9 using NaOH is performed as an alternative to preservation using Ammonium Sulfate Buffer.

14.2. Hach ChromaVer 3 combined reagent is used in place of laboratory prepared H_2SO_4 solution and Diphenylcarbazide solution.

14.3. Calibration standards are purchased as certified standards.

15. Pollution Prevention and Waste Management

15.1. Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling or other appropriate SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

15.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16. References

16.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision of methods 7196A and 3060A.

16.2. Standard Methods for the Examination of Waste and Wastewater, Method 3500-Cr B.

16.3. 40 CFR, Part 136, Table IB and Table II, Method Update Release, 2012

16.4. Pace Analytical Quality Manual; latest revision.

16.5. TNI Standard; Quality Systems section; latest revision.

17. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1. Not Applicable

18. Revisions

Document Number	Reason for Change	Date
S-IN-I-063-rev.9	1. Table 7.1: revised hold time for soils. 2. Section 9.2.2.3: adjusted wording to match actual practice. 3. Section 10.2: added equation for correlation coefficient. 4. Sections 10.3 and 10.5: added sentence to end of paragraph about rerunning samples. 5. Section 10.4: added "preceding or" to language about bracketing CCVs. 6. General: updated all SOP references 7. Removed sentence from section 13.2 about regulatory limits. 8. Added soil equation to section 12.5. 9. Replaced Whatman filter with 0.45um filter in sections 9.4 and 12.1 10. Reworded sections 12.2 and 12.3 to reflect current practices. 11. Added paragraph to section 13.5 about soil spikes. 12. Added 3060 phase diagram to section 17.	13Nov2007

S-IN-I-063-rev.10	1. Cover page converted to periodic review format and added SM3500-Cr D reference. 2. Table of Contents fixed to include sections 10 and 11. 3. Section 1: added SM3500-Cr D reference. 4. Section 3: added language that limits, volumes, vendors, are subject to change. 5. Table 7.1: updated to clarify storage and separated requirements for unpreserved and preserved aqueous samples. 6. Section 8: updated and added "or equivalent" where applicable. 7. Section 9: updated and clarified separate standards as intermediate and working. Created a table for working calibration standard examples. 8. Section 10: completely revised to individually clarify calibration requirement including corrective actions. 9. Section 11: clarified procedure including intended run sequence and added calculations for final concentration. 10. Section 12: updated limits and corrective actions and added instructions for aqueous batch QC preparation. 11. Section 13: removed SOP references. 12. Section 15: changed NELAC to TNI and added SM3500-Cr D reference.	23Jun2011
S-IN-I-063-rev.10	1. Cover: revised Standard Methods reference from "D" to "B". 2. Section 1.1: revised Standard Methods reference and added a note that SM 3500-Cr B supersedes and is equivalent to SM 3500-Cr D. 3. Section 3.1: added a reference to the alkaline digestion SOP for soil procedures. 4. Section 3.2: added reference to LIMS for MDLs. 5. Table 7.1: updated for water preservation procedure and changed preserved water holding time from 30 days to 28 days. 6. Table 9.1: added reagents for Ammonium Sulfate Buffer 7. Section 9.2.3: added a reference to the alkaline digestion SOP for reagents and standards associated with the preparation of solid samples. 8. Section 11.7: added requirement to qualify over range results 9. Section 11.8: revised/fixed calculation for concentration from curve. 10. Table 12.1: revised method blank corrective actions and added references to pH and Eh procedures for solid sample MS/MSD corrective action. 11. Section 12: added/expanded aqueous LCS preparation. 12. Inserted new Method Modifications section. 13. References: revised Standard Methods reference and added reference to 40 CFR 2012 MUR.	26Sep2012

STANDARD OPERATING PROCEDURE

THE DETERMINATION OF VOLATILE ORGANICS BY GC/MS

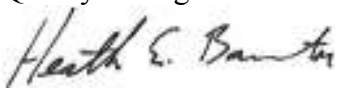
REFERENCE METHOD: EPA SW-846 METHODS 8260B, 5030A, 5030B AND 5035A

LOCAL SOP NUMBER:	S-IN-O-029-rev.17
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-O-029-rev.16

LOCAL APPROVAL

General Manager — September 25, 2012 Date

Quality Manager — September 25, 2012 Date



Department Manager — September 24, 2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

1.1. This Standard Operating Procedure (SOP) documents the procedures used by Pace Analytical Services – Indianapolis to determine the concentration of Volatile Organic Compounds (VOCs) in environmental samples. The laboratory utilizes purge-and-trap GC/MS and bases these documented procedures on those listed in SW-846 Method 8260B, 5030A, 5030B, and 5035A.

2. Summary of Method

2.1. Volatile organic compounds are introduced into the gas chromatograph by a purge-and trap method. The analytes are purged from a sample aliquot or extract using an inert gas. The purged analytes are collected on an absorbent trap. At the completion of the purge time, the trap is rapidly heated and back flushed to drive out the trapped analytes. The analytes are transferred into the inlet of a capillary gas chromatography column. The carrier gas flow through the column is controlled and the temperature is increased according to a set program to achieve optimum separation of purged analytes. The mass spectrometer is operated in a repetitive scan mode. Analytes are identified by the GC/MS retention times and by a comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected primary ion relative to an internal standard against a calibration curve.

3. Scope and Application

3.1. This method is applicable to most organic compounds that have boiling points below 200 ºC and are insoluble or slightly soluble in water. Volatile water-soluble compounds may also be determined although quantitation limits are typically higher due to their hydrophilic properties (e.g. ketones, oxygenates). The list of compounds and reporting limits is found in Table 1. Refer to the LIMS for method detection limits.

3.2. This method is applicable to most water and solid samples, regardless of moisture content. Matrices are groundwater, surface water, soil, sediment and waste. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interfere with sample analysis.

3.3. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the absorbent trap. The use of polytetrafluoroethylene (PTFE, Teflon) as thread sealants, tubing, or in flow controllers is highly recommended since other materials can be sources of contamination which may concentrate in the trap during the purging.

4.2. A common source of interfering contamination is carryover. This may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive action to this condition is rinsing the purging apparatus and sample syringes with organic free water between samples. Analyze one or more blanks to check for contamination prior to sample analysis.

4.3. Since methylene chloride and acetone are common laboratory solvents, special precautions must be taken. The volatiles analysis and sample storage area must be located as far as possible from areas where these solvents are used or stored. Where possible, the volatiles analysis and sample storage area should be served by a separate HVAC system and maintained under positive pressure to prevent intrusion of contaminants. Laboratory clothing previously exposed to methylene chloride fumes during extraction procedures can contribute to sample contamination.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2. Samples

Take precautions when handling samples. Samples must always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

5.3. Equipment

Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments must be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones.

The purge and trap concentrator and autosampler use gas under pressure to purge samples and, in some cases, drive the robotic assemblies. These high pressures introduce the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are required at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
5030B Aqueous	Minimum (3) VOA vials Additional sample is required if MS/MSD is requested	Acidified w/ 1:1 HCl to pH<2, no headspace	0 ° to 6°C	pH>2: Analysis must be completed within 7 days of collection date. pH <2: Analysis must be completed within 14 days of collection date. (pH determined post analysis)
5035A Solid Terra Core Kits (Preferred)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) 5-g portions in vials with magnetic stir bar and 5.0mL reagent water plus one (1) 5 g portion in a vial with 5.0mL methanol. Additional sample is required if MS/MSD is requested.	No preservative or Methanol	0 ° to 6°C for no more than 48 hours from collection then freeze at -7°C to -20°C.	Analysis must be completed within 48 hours if samples are not frozen or preserved with methanol prior to the expiration of the 48 hour period. The holding time may be extended to 14 days if the sample is frozen or preserved with methanol prior to the expiration of the 48 hour period.
5035A Solid Coring Devices (Alternate)	One (1) 2-4 oz. wide mouth jar for % moisture **AND** Two (2) EnCore, TerraCore or similar sampling tubes. Additional sample is required if MS/MSD is requested.	No preservative Sample is extruded into a vial with a magnetic stir bar and 5.0mL reagent water.	Freeze at -7°C to -20°C within 48 hours of collection.	Analysis must be completed within 14 days of collection date.
5030A Solid Bulk Jars	One (1) 2-4 oz. wide mouth jar for % moisture **AND** One bulk sample jar, usually 4 oz. or 8oz.	No preservative Sample is weighed into a vial with a magnetic stir bar and 5.0mL reagent water.	0 ° to 6°C	Analysis must be completed within 14 days of collection date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatographs	Agilent	Lab uses models 5890, 6850 and 6890	Or equivalent system
P&T Concentrators	EST Analytical or Tekmar	Lab uses Tekmar 3000 series, Encons, and Encon Evolution	Or equivalent system
Data Systems	Agilent	Chemstation	Or equivalent system
Autosamplers	EST Analytical	Lab uses EST 8100s, Centurion, and Centurion WS	Or equivalent system
Mass Spectrometers	Agilent	Lab uses models 5971, 5973 and 5975	Or equivalent system

8.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Agilent	J&W Scientific DB-624	20m x 0.18mm x 1um or equivalent
Trap	Supelco	Trap K	Or equivalent

8.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials	Supelco or equivalent
Analytical balance	Able to measure to nearest 0.1g	Ohaus or Sartorius or equivalent
Sample vials	40mL vials; pre-cleaned	Eagle Picher or equivalent

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	ASTM Type II water
Methanol	Purge-and trap grade or equivalent
Sand	Or equivalent to be used as a simulated soil matrix.

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.1 Standard Definitions

Standard	Description	Comments
Tune Standard	4-Bromofluorobenzene (BFB) solution used to verify ion response ratios prior to analysis	Must inject between 5 and 50ng
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains required spiking compounds, at a minimum, and is used to prepare MS/MSD sets.	Same solution can be used for the LCS and MS/MSD.

9.2.2. Storage Conditions

Table 9.2 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock VOA calibration standards	Restek; catalog # 558679; 250-5000ug/mL and #554123; 500ug/mL and #30216; 2000ug/mL and #30006; 5000ug/mL or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Stock Gas calibration standards	Restek; catalog # 30439; 200ug/mL and o2si; catalog#020229-09, 10,000ug/mL or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Intermediate VOA calibration standard	Refer to Section 9.2.3.1.	Solution good for 1 month from preparation	Same as stock standard.
Intermediate Gas calibration standard	Refer to Section 9.2.3.2.	Solution good for 1 week from preparation	Same as stock standard
Working VOA calibration standards	Refer to Section 9.2.3.3.	One-time use	Not applicable
Stock VOA ICV/Spiking standards	o2si; catalog #120868-02; 250-5000ug/mL and #020201-01, 1000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Intermediate VOA ICV/Spiking standard	Refer to Section 9.2.3.4.	Solution good for 1 month from preparation	Same as stock standard
Working ICV/Spiking standard	Refer to Section 9.2.3.5.	One-time use	Not applicable
Stock VOATune/ Surrogate standard	Restek; catalog #30240, 2500ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Stock VOA Internal standards	Restek; catalog #30241, 2500ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working Tune/Surrogate/Intern al standard mix	Refer to Section 9.2.3.7.	Solution good for 1 month from preparation	Stored on autosampler under pressure in a 5mL vial.

9.2.3. Preparation Procedures

9.2.3.1. Intermediate VOA Calibration Standard Preparation

Dilute 500uL of Restek #558679 plus 500uL of Restek #554123 plus 250uL of Restek #30216 plus 125uL of Restek #30006 to 2.5mL with Methanol for a final nominal concentration of 50mg/L.

9.2.3.2. Intermediate Gas Calibration Standard Preparation

Dilute 500uL of Restek #30439 plus 200uL of o2si #020229-09 to 2.0mL with Methanol for a final nominal concentration of 50mg/L.

9.2.3.3. Working Calibration Standards Preparation

Refer to Table 9.3 for examples of possible one-time use calibration standards.

Table 9.3 – Working Calibration Standards (examples only)

Standard	Int. Calibration Standard amount	Int. Gas Standard amount	Final Total Volume	Final Concentration
Calibration Std 1	1uL	1uL	50mL	1ppb
Calibration Std 2	2uL	2uL	50mL	2ppb
Calibration Std 3	5uL	5uL	50mL	5ppb
Calibration Std 4	10uL	10uL	50mL	10ppb
Calibration Std 5	2uL	2uL	5mL	20ppb
Calibration Std 6 (CCV)	5uL	5uL	5mL	50ppb
Calibration Std 7	15uL	15uL	5mL	150ppb
Calibration Std 8	30uL	30uL	5mL	300ppb

9.2.3.4. Intermediate VOA ICV/Spiking Standard Preparation

Dilute 200uL of o2si #120868-02 plus 200uL of o2si #020201-01 to 2mL with Methanol for a final nominal concentration of 50mg/L.

9.2.3.5. Working ICV/Spiking Standard Preparation

Add 50uL of the Intermediate ICV standard per 50mL water for a final ICV concentration of 50ug/L.

9.2.3.6. Laboratory Control Sample (LCS) and Matrix Spike (MS/MSD) Preparation

9.2.3.6.1 **Aqueous LCS:** add 5uL of the Intermediate ICV/Spiking standard to 5mL reagent water for an LCS concentration of 50ug/L.

9.2.3.6.2 **Aqueous MS:** add 5uL of the Intermediate ICV/Spiking standard to 5mL sample for an MS concentration of 50ug/L.

9.2.3.6.3 **Soil LCS:** place approximately 5g of simulated soil matrix and 5mL reagent water into a vial. Add 5uL of the Intermediate ICV/Spiking standard for an LCS concentration of 50ug/Kg.

9.2.3.6.4 **Soil MS:** place approximately 5g of sample and 5mL reagent water into a vial. Add 5uL of the Intermediate ICV/Spiking standard for an LCS concentration of 50ug/Kg.

9.2.3.7. Working Tune/Surrogate/Internal Standard Preparation

Dilute 100uL of Restek #30240 plus 100uL of Restek #30241 to 5mL with Methanol for a final concentration of 50mg/L.

10. Calibration

10.1. Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned by injecting 5-50ng BFB. This is done by analyzing a standard containing BFB. The tune verification standard can be combined with the CCV standard provided that the amount of BFB introduced into the system meets the criteria. Use the BFB mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected.

Mass (m/z)	Ion Abundance criteria
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	<2% of m/z 174
174	>50% of m/z 95
175	5 to 9% of m/z 174
176	95 to 101% of m/z 174
177	5 to 9% of m/z 176

If the ratios do not meet the criteria above, reanalyze the BFB tune. If the BFB still fails the criteria, instrument maintenance and/or preparation of new standards must be considered.

10.2. Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

10.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

10.4. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

10.5. The %RSD should be should be ≤15% for each target analyte. However, the %RSD for each individual Calibration Check Compound (CCC) must be ≤30%. If an RSD is >30% for any CCC, then instrument maintenance and/or preparation of new standards must be considered before attempting recalibration. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl Chloride

10.6. System Performance Check Compounds (SPCCs) are checked for a minimum average response factor (RF_{avg}) to determine potential instability and/or degradation caused by contaminated lines or active sites in the system. The minimum RF_{avg} for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

10.7. If the percent relative standard deviation (%RSD) of the RFs for a compound is ≤15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

10.8. If the % RSD for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.9. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$r = \frac{\sum_{i=1}^{N} (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} (Y_i - \overline{Y})^2\right)}}$$

10.10. Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be used and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be $\geq$0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$\text{COD} = \frac{\sum_{i=1}^{N} (Y_i - \overline{Y})^2 - (\frac{n-1}{n-p}) \sum_{i=1}^{N} (Y_i - Y_i')^2}{\ldots\ldots\ldots\ldots \sum_{i=1}^{N} (Y_i - \overline{Y})^2}$$

10.11. Initial Calibration Corrective Action: If the initial calibration does not meet the required criteria to be used for quantitative purposes, a new initial calibration must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.12. Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent difference (%D) of the observed ICV response from the initial calibration average response, according to the following equation where RF_v is the response factor of the verification standard:

$$\% \text{ Difference } (\%D) = [RF_v - RF_{avg}] / RF_{avg} * 100$$

The ICV is analyzed immediately following the initial calibration curve. All compounds must have a maximum percent difference of +/- 30% from the initial calibration curve. Allowances for poor performers (ie, Acrolein, Ketones, 2 CEVE, etc) must be approved by the department manager and clearly documented as to which compounds are affected. **For BP, the ICV must meet the CCV criteria.**

10.13. ICV Corrective Action: If the ICV fails the criteria, another ICV may be analyzed. If the second ICV fails, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV fails during an overnight run and is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

10.14. Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a BFB tune that must meet the criteria in Section 10.1, followed by a Continuing Calibration Verification (CCV) standard. The CCV is normally prepared using the same standard solution used for the initial calibration but an ICV/LCS can be used as a CCV if it passes the required criteria for a CCV.

10.15. The % difference (%D) between initial calibration and CCV response factors for CCCs must be ≤20%. The response factors for all SPCCs in the CCV standard must meet the criteria in Section 10.6. An LCS may be used as a CCV provided that it passes the CCV acceptance criteria. **For BP, surrogate compounds must also be ≤20%D.**

10.16. The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

10.17. CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11. Procedures

11.1. Configure the purge & trap system and GC/MS system per manufacturer's instructions. All samples must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

11.2. Sample Preparation and Handling

11.2.1. Aqueous Samples

Water samples to be analyzed using the Centurion autosampler require no sample preparation and are loaded as full 40mL VOA vials, unless they require a dilution. Refer to Section 7 for additional information regarding sample handling.

Water samples to be purged on the Archon/8100 autosampler are prepared by quickly measuring a 5mL aliquot of the sample using a 5mL gastight syringe and transferring it to a 40mL VOA vial. This is done as quickly as possible to minimize analyte loss. The syringe is thoroughly rinsed inside and out with reagent water before measuring each sample.

Dilutions on aqueous samples must be prepared in a volumetric fashion. Sample aliquots are measured in either a volumetric pipet or gas-tight syringe and brought to volume in either a volumetric flask or gas-tight syringe.

After analysis, check the residue in the vial using pH paper. The pH should be <2. Holding time for water samples with pH >2 is 7 days. Appropriately footnote on the sequence log and in LIMS any sample not meeting the pH requirement and/or holding time requirement. A stamp may be used to document on sequence logs that all water samples are pH<2 unless otherwise noted.

11.2.2 Soil Samples

11.2.2.1 Low-Level soils

Preferably, samples received for low level analysis should be contained in pre-weighed Terra Core vials with Organic Free Water (OFW). Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial to determine the sample weight. The sample is ready for analysis. Refer to Section 7 for additional information regarding sample handling.

Alternatively, samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial either with or without 5mL OFW and a magnetic stir bar. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight

determined initially to determine the sample weight. The sample is ready for analysis.

Samples received in bulk soil jars are applicable to Method 8260A only and are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add approximately 5g of the sample to the vial. Record the sample weight. Add 5mL of reagent water and a magnetic stir bar and cap the vial. The sample is ready for analysis.

11.2.2.2 Medium-Level soils

Preferably, samples received for medium-level analysis should be received in pre-weighed Terra Core vials with methanol as a preservative. Prior to analysis the sample weight must be determined and recorded by weighing the vial and recording the weight. Subtract the tare weight indicated on the vial to determine the sample weight. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL OFW is used for analysis. Refer to Section 7 for additional information regarding sample handling.

Alternatively, samples received in coring devices, such as Encore, must be extruded into a pre-weighed VOA vial with 5mL methanol. Record the weight of the vial after the sample has been placed into it. Subtract the tare weight determined initially to determine the sample weight. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL OFW is used for analysis.

Samples received in bulk soil jars are applicable to Method 8260A only and are sub-sampled into a VOA vial. Place an empty VOA vial on the balance pan and tare the balance. Quickly add approximately 5g of the sample to the vial. Record the sample weight. Add 5mL methanol and cap the vial. The sample is mixed well on a vortex mixer and allowed to settle. A maximum of 200uL of the methanol extract per 5mL OFW is used for analysis.

11.3 Qualitative Analysis

11.3.2 The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

11.3.3 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for the characteristic ions.

11.3.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.4 Quantitative Analysis: Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. See Sections 11.6, 11.7, and 11.8 for equations to calculate the amount of analyte or surrogate introduced into the instrument. Calculations are subject to change based on the data reduction software used.

11.5 When the average response factor from the initial calibration is used, calculate the on-column amount:

$$X_s = (A_s/RF_{avg}) \times (C_{is}/A_{is})$$

Where:
X_s = On-column concentration of the analyte or surrogate
A_s = Peak area or height of the analyte or surrogate in the sample
A_{is} = Peak area or heath of the internal standard in the sample
C_{is} = Concentration of the internal standard in the sample
RF_{avg} = The average response factor from the initial calibration

11.6 When a linear curve fit is used, calculate the on-column amount:

$$X_s = \frac{[A_s/A_{is} - b]}{a} \times C_{is}$$

where: A_s = Peak area or height of the analyte or surrogate in the sample
A_{is} = Peak area or height of the internal standard in the sample
X_s = On-column concentration of the analyte or surrogate
C_{is} = Concentration of the internal standard in the sample
a = Slope of the line
b = The intercept

11.7 When a non-linear or quadratic curve fit is used, calculate the on-column amount:

$$X_s = \frac{-b + \sqrt{b^2 - 4a\left(c - \frac{A_s}{A_{is}}\right)}}{2a} \times C_{is}$$

where: X_s = On-column concentration of the analyte or surrogate
A_s = Peak area or height of the analyte or surrogate in the sample
A_{is} = Peak area or height of the internal standard in the sample
C_{is} = Concentration of the internal standard in the sample
a = Slope of the line
b = The intercept
c = coefficient of the polynomial

11.8 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_t)(D)}{(V_i)(V_s)} \qquad \text{Solid Sample (ug/Kg)} = \frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$$

Where: X_s = On-column concentration of the analyte in the sample aliquot injected
V_t = Total volume of concentrated extract
D = Dilution factor
V_i = Volume of the extract injected in uL
V_s = Volume of aqueous sample extracted in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{(100 - \%\text{Moisture})} \times 100$$

12 Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Reanalyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	Reanalyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
For BP only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed at the same dilution or more concentrated. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

12.2 Method Blank Preparation

12.2.1 Waters on Archon autosamplers: The Method Blank consists of a 40mL VOA vial containing 5mL reagent water.

12.2.2 Waters on Centurion autosamplers: The Method Blank consists of a 40mL VOA vial filled completely with reagent water.

12.2.3 Low-level soils: The Method Blank consists of a 40mL VOA vial containing approximately 5g simulated soil matrix and 5mL reagent water.

12.2.4 Medium-level soils: The Method Blank consists of a 40mL VOA vial containing 5mL reagent water and 200uL methanol.

12.3 Laboratory Control Sample (LCS) and Matrix Spike (MS/MSD) Preparation: Refer to Section 9.2.3.6.

12.4 Allowable Marginal Exceedances: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: For BP, the LCS shall be allowed to be outside the control limits but ≥10% for up to four additional volatile compounds with the exception of benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, total xylenes and any requested oxygenate without corrective action.

12.5 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.6 MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.7 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13 Method Performance

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted annually for each matrix per instrument.

13.2 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14 Method Modifications

14.1 GC columns and chromatographic conditions may differ from those recommended.

14.2 Calibration solutions are purchased as certified standards.

15 Pollution Prevention and Waste Management

15.1 Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

15.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B, 8000C, 8260A, 8260B, 5030A, 5030B, and 5035A.

16.2 Pace Analytical Quality Manual; latest revision.

16.3 TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1 Table 1: Method 8260B Target Compounds and Reporting Limits
17.2 Table 2: Characteristic Ions of Target Compounds

18 Revisions

Document Number	Reason for Change	Date
S-IN-O-029-rev.12	1. Added new section 11.6.2 regarding signal-to-noise ratio. 2. Added new section 10.2.3 regarding when and how to drop points from an initial calibration curve (copied from Quality Manual). 3. Added new section 10.5 regarding the inability to use Grand Mean Averaging. 4. Section 10.2.2: added last paragraph stating why the lab does not curve its surrogate compounds. 5. Table 11.1: revised EPIC Pro instrument names	11Sep2007
S-IN-O-029-rev.13	1. Table 10.2: revised internal standard retention time limits (for BP samples only) and added line for client samples regarding internal standard acceptance and that BP samples that have failing internal standards must be rerun. Updated ICV acceptance criteria (same as CCV). 2. Section 11.4.1.1: added language that all water samples must have the pH measured and recorded. 3. Section 9.2.4.4: adjusted recipe for surrogates per HEB. 4. Section 10.3: changed 'direct injecting' to 'purging'. 5. Table 11.1: added new instrument (50MSV4) and removed old naming convention. 6. Section 11.4.1.3.2: revised maximum amount of methanol to 200uL.	11Mar2008
S-IN-O-029-rev.14	1. Section 2: changed helium to nitrogen purge gas. 2. Table 9.2: changed gas standard expiration to 1 month. 3. Table 10.2: revised ICV and CCV criteria 4. Table 11.1:removed specific instrument IDs – made generic. 5. Section 11.4: added pre-screening procedure. 6. Section 11.4.1: added requirement that samples that are pH>2 and analyzed after 7 days be qualified. 7. Section 11.4.1.2.1: removed requirement that soil subsampling be done using an Encore sampling device. 8. Table 12.1: revised LCS/MS compounds and criteria.	31Mar2009
S-IN-O-029-rev.15	1 Removed SOP template from cover page 2 Section 6: removed SOP specific definitions 3 Table 7.1: replaced with a more detailed table. 4 Section 8: added "or equivalent" where applicable 5 Section 9.2.4: added wording regarding expiration dates. 6 Table 10.1: removed 7 Section 10.3.3: clarified RL wording 8 Table 10.2: added use of LCS as CCV 9 Section 10.5.3: added use of LCS as CCV 10 Section 11.1.1: revised to consult manufacturer's instructions 11 Table 11.1: removed 12 Section 11.3: removed Note 13 Section 11.4.2.1: improved wording regarding samples pH 14 Section 11.4.2.2: replaced "weight" with "mass" 15 Section 11.4.2.3.2.1: added use of gastight syringe 16 Table 12.2: reworded exceptions for clarity 17 Table 12.3: reworded IS Area criteria for clarity	12Mar2010

S-IN-O-029-rev.16	1. Section 3: added language that limits, volumes, vendors, are subject to change. 2. Table 7.1: updated to clarify hold time and acceptable preservation. Deleted Table 7.2. 3. Section 8: updated and added "or equivalent" where applicable. 4. Section 9: updated and clarified separate standards as intermediate and working. Created a table for all calibration standards 5. Section 10: completely revised to individually clarify calibration requirement including corrective actions. Simplified BFB tune information. 6. Section 11: clarified procedure including calibration calculations and added calculations for final concentration. Removed information about TICs. 7. Section 12: updated information and corrective actions and added instructions for batch QC preparation. Clarified marginal exceedance criteria. 8. Section 13: removed SOP references. 9. Section 15: changed NELAC to TNI. 10. Attachments: removed previous Att. 1 and added Att. 1 and Att. 2.	13Jun2011
S-IN-O-029-rev.16	1. Section 3.1: added reference to MDLs 2. Section 9.1: added simulated soil matrix. 3. Section 9.2.3.6: added detail for preparation of LCS and MS to include simulated soil matrix. 4. Table 12.1: revised method blank corrective action. 5. Section 12.2.3: revised low-level soil method blank prep to include simulated soil matrix. 6. Inserted new Method Modifications section.	24Sep2012

Table 1: Method 8260B Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L)	RL soil Low-level (ug/kg)	RL soil Medium-level (ug/kg)
Dichlorodifluoromethane	5	5	125
Chloromethane	5	5	125
Vinyl Chloride	2	5	125
Bromomethane	5	5	125
Chloroethane	5	5	125
Trichlorofluoromethane	5	5	125
Methylene Chloride	5	20	500
1,1-Dichloroethene	5	5	125
trans-1,2-Dichloroethene	5	5	125
1,1-Dichloroethane	5	5	125
2,2-Dichloropropane	5	5	125
cis-1,2-Dichloroethene	5	5	125
Chloroform	5	5	125
Bromochloromethane	5	5	125
1,1,1-Trichloroethane	5	5	125
Carbon Tetrachloride	5	5	125
1,1-Dichloropropene	5	5	125
Benzene	5	5	125
1,2-Dichloroethane	5	5	125
Trichloroethene	5	5	125
1,2-Dichloropropane	5	5	125
Bromodichloromethane	5	5	125
Dibromomethane	5	5	125
Toluene	5	5	125
1,1,2-Trichloroethane	5	5	125
Tetrachloroethene	5	5	125
1,3-Dichloropropane	5	5	125
Dibromochloromethane (Chlorodibromomethane)	5	5	125
1,2-Dibromoethane (EDB)	5	5	125
Chlorobenzene	5	5	125
1,1,1,2-Tetrachloroethane	5	5	125
Ethylbenzene	5	5	125
m&p-Xylene	5	5	125
o-Xylene	5	5	125
Styrene	5	5	125
Bromoform	5	5	125
Isopropylbenzene	5	5	125
1,1,2,2-Tetrachloroethane	5	5	125
Bromobenzene	5	5	125
1,2,3-Trichloropropane	5	5	125
n-Propylbenzene	5	5	125
2-Chlorotoluene	5	5	125
1,3,5-Trimethylbenzene	5	5	125
4-Chlorotoluene	5	5	125
1,2,4-Trimethylbenzene	5	5	125
sec-Butylbenzene	5	5	125
tert-Butylbenzene	5	5	125
p-Isopropyltoluene	5	5	125
1,3-Dichlorobenzene	5	5	125
1,4-Dichlorobenzene	5	5	125

Analyte	RL water (ug/L)	RL soil Low-level (ug/kg)	RL soil Medium-level (ug/kg)
n-Butylbenzene	5	5	125
1,2-Dichlorobenzene	5	5	125
1,2,4-Trichlorobenzene	5	5	125
Hexachlorobutadiene	5	5	125
Naphthalene	5	5	125
1,2,3-Trichlorobenzene	5	5	125
trans-1,3-Dichloropropene	5	5	125
cis-1,3-Dichloropropene	5	5	125
Acetone	100	100	2500
2-Butanone (MEK)	25	25	625
4-Methyl-2-pentanone (MIBK)	25	25	625
Acrolein	50	100	2500
Acrylonitrile	100	100	2500
2-Hexanone	25	100	2500
Vinyl Acetate	10	100	2500
Iodomethane	10	100	2500
Methyl tert-butyl ether	4	5	125
Carbon Disulfide	10	10	250
trans-1,4-Dichloro-2-butene	100	100	2500
Ethyl Methacrylate	100	100	2500

[1]Target Compounds and Reporting Limits are subject to change.

Table 2: Characteristic Ions of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
Trichlorofluoromethane	101	103
Methylene Chloride	84	86, 49
1,1-Dichloroethene	96	61, 63
trans-1,2-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83
2,2-Dichloropropane	77	97
cis-1,2-Dichloroethene	96	61, 98
Chloroform	83	85
Bromochloromethane	49	128
1,1,1-Trichloroethane	97	99, 61
Carbon Tetrachloride	117	119, 121
1,1-Dichloropropene	75	110, 77
Benzene	78	52, 77
1,2-Dichloroethane	62	98, 64
Trichloroethene	95	97, 130, 132
1,2-Dichloropropane	63	62, 112
Bromodichloromethane	83	85, 127
Dibromomethane	93	95, 174
Toluene	91	92
1,1,2-Trichloroethane	83	97, 85
Tetrachloroethene	166	129, 168
1,3-Dichloropropane	76	78
Dibromochloromethane (Chlorodibromomethane)	129	127
1,2-Dibromoethane (EDB)	107	109
Chlorobenzene	112	77, 114
1,1,1,2-Tetrachloroethane	131	133, 119
Ethylbenzene	106	91
m&p-Xylene	106	91
o-Xylene	106	91
Styrene	104	78
Bromoform	173	175, 254
Isopropylbenzene	105	120
1,1,2,2-Tetrachloroethane	83	131, 85
Bromobenzene	77	156, 158
1,2,3-Trichloropropane	75	77, 110
n-Propylbenzene	91	120
2-Chlorotoluene	91	126
1,3,5-Trimethylbenzene	105	120
4-Chlorotoluene	126	91
1,2,4-Trimethylbenzene	105	120
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
p-Isopropyltoluene	119	134, 91
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
n-Butylbenzene	91	92, 134

Analyte	Primary Ion	Secondary Ion(s)
1,2-Dichlorobenzene	146	111, 148
1,2,4-Trichlorobenzene	180	182, 145
Hexachlorobutadiene	225	223, 227
Naphthalene	128	127
1,2,3-Trichlorobenzene	180	182, 145
trans-1,3-Dichloropropene	75	77
cis-1,3-Dichloropropene	75	77
Acetone	43	58
2-Butanone (MEK)	43	57, 72
4-Methyl-2-pentanone (MIBK)	43	58, 85
Acrolein	56	55
Acrylonitrile	53	52, 51
2-Hexanone	43	58, 100
Vinyl Acetate	43	86
Iodomethane	142	127
Methyl tert-butyl ether	73	57
Carbon Disulfide	76	78
trans-1,4-Dichloro-2-butene	53	88, 75
Ethyl Methacrylate	69	99, 114
Surrogates	Primary Ion	Secondary Ion(s)
Dibromofluoromethane	113	111
Toluene-d8	98	99, 100
4-Bromofluorobenzene	95	174, 176
Internal Standards	Primary Ion	Secondary Ion(s)
Fluorobenzene	96	-
Chlorobenzene-d5	117	82, 119
1,4-Dichlorobenzene-d4	152	115, 150

[2]Target compounds subject to change.

STANDARD OPERATING PROCEDURE

THE DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS)

REFERENCE METHOD: EPA SW-846 METHOD 8082 AND EPA METHOD 608

SOP NUMBER:	S-IN-O-050-rev.11
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-O-050-rev.10

APPROVAL

Karl Anderson
General Manager — April 20, 2012 Date

Bert Schlaye
Quality Manager — April 11, 2012 Date

Steven R Long
Department Manager — April 11, 2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of polychlorinated biphenyls (PCBs) in aqueous and solid samples, including oils and wipes, while meeting the requirements specified in EPA methods 8082 and 608.

2. Summary of Method

2.1. The samples are extracted using Method 3546, Microwave Extraction for soils and Method 3510C, Separatory Funnel Extraction for waters, or other appropriate technique or solvents.

2.2. Cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes.

2.3. After cleanup, the extract is analyzed by a gas chromatograph fitted with an electron capture detector (ECD). Aroclor identification is confirmed by a secondary column.

3. Scope and Application

3.1. This SOP is used to determine the concentrations of PCBs as Aroclors in extracts from solid, aqueous, oil and wipe matrices. The target Aroclors are listed below:

Aroclor	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

3.2. Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns than those of Aroclor standards.

3.3. Reporting limits are 0.5ug/L for waters, 35ug/Kg for soils, 1mg/Kg for oils and 0.5 total ug for wipes. Reporting limits may vary as a function of volume or weight of sample extracted and extract final volume.

3.4. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.5. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC systems and interpretation of GC PCB data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Matrix interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware. These interferences lead to discrete artifacts or elevated baselines in gas chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates, which are easily extracted during lab operations. Avoiding the use of plastics in the lab can best minimize interferences from phthalates.

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 site being sampled. Cleanup procedures may be used to remove such interferences.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock PCB standards are toxic and should be handled with extreme care.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves**,** lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood. **NOTE: All sample waste and waste products from PCB extraction and analysis must be stored in a separate satellite waste container, which shall remain closed unless waste is being actively added.**

5.3. Equipment

Portions of the preparation and analytical equipment operate at high temperatures. Care must be taken to minimize accidents and injuries when working on or with this equipment. Equipment should be turned off or the temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on equipment.

6. Definitions

6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	1 liter in amber glass bottle	None required	0°C to 6°C	Extract within 7 days of collection and analyze within 40 days of extraction
Solid	>100 grams in 4 or 8oz glass jar	None required	0°C to 6°C	Extract within 14 days of collection and analyze within 40 days of extraction
Wipes	One wipe per glass container	None required	0°C to 6°C	Extract within 14 days of collection and analyze within 40 days of extraction
Oils	Glass container	None required	0°C to 6°C	Extract within 14 days of collection and analyze within 40 days of extraction

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
GC	Agilent	7890 with LTM module or equivalent	Equipped with dual ECD Detectors; dual columns, and dual injectors, or equivalent system.
Autosampler	Agilent	7673A or equivalent	100 position
Data System	Chemstation acquisition; Target integrations		Or equivalent software
Primary Column	Restek	DB5-MS LTM or equivalent	Fused silica; 15 meters, 0.32mm ID, 0.50um film thickness, or equivalent column
Secondary Column	Restek	DB-1701 LTM or equivalent	Fused silica; 15 meters, 0.32mm ID, 0.25um film thickness, or equivalent column

8.2. General Supplies

Item	Vendor	Description
Glass syringes	Hamilton or equivalent	Various sizes
Glass vials	Fisher or equivalent	20mL volume with Teflon lined caps
Autosampler vials	Fisher or equivalent	2mL volume with Teflon lined crimp tops
Micro-inserts	Hewlett Packard or equivalent	Various sizes

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
n-Hexane	Pesticide or reagent grade or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	ICAL
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	CCV
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the calibration curve.	ICV
Spiking Standard	This solution contains method specified analytes and is used to spike LCS, MS and MSD.	Same solution can be used for the LCS and MS/MSD

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock PCB calibration standards	Restek; 1016 catalog # 32006, 1260 catalog # 32012, 1221 catalog #32007 and 1232 catalog #32008 each at 1000ug/mL in hexane, or equivalent. Ultra: 1242; catalog # PP-311; AR 1248; catalog # PP-341-1; 1254; catalog # PP-350 each at 100ug/mL in hexane, or equivalent. Other Aroclor standards may be used.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate PCB calibration standards	Refer to Section 9.2.3.1	Solution good for 6 months from preparation.	Same as stock standard.
Working PCB calibration standards	Refer to Section 9.2.3.2	Solution good for 6 months from preparation.	Same as stock standard.
Stock PCB ICV standard	Ultra; AR 1016; catalog # PP-281-1 and AR 1260; catalog # PP-361-1 each at 100ug/mL in hexane, or equivalent. Other Aroclor standards may be used.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate PCB ICV standard	Refer to Section 9.2.3.3	Solution good for 6 months from preparation.	Same as stock standard.
Working PCB ICV standard	Refer to Section 9.2.3.4	Solution good for 6 months from preparation.	Same as stock standard.
Stock TCMX surrogate standard	Restek; catalog # 32027; 2,4,5,6-tetrachloro-m-xylene; 200ug/mL in acetone, or equivalent.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions

Working TCMX surrogate standard	Refer to Section 9.2.3.5	Solution good for 6 months from preparation.	Same as stock standard.
Intermediate Single Point PCB Calibration Standards	Refer to Section 9.2.3.6	Solution good for 6 months from preparation.	Same as stock standard.
Working Single Point PCB Calibration Standards	Refer to Section 9.2.3.7	Solution good for 6 months from preparation.	Same as stock standard.

9.2.3. Standard Preparation Procedures

9.2.3.1. Intermediate PCB Calibration Standard Preparation

Dilute 10uL of the stock Aroclor 1016 standard (1000ug/mL), 10uL of the stock Aroclor 1260 standard (1000ug/mL) and 50uL of the Stock TCMX surrogate standard (200ug/mL) to 1mL with hexane to produce a final concentration of 10ug/mL for each component. Intermediate Calibration Standards of other Aroclors can be prepared using this formula to facilitate the preparation of a calibration curve for an individual Aroclor other than 1016 or 1260.

9.2.3.2. Working PCB Calibration Standard Preparation

The following are examples of calibration standard concentrations and could vary based on requirements:

Standard	Intermediate Standard amount	Final Volume in Hexane	Final PCB/Surr Concentration
Standard 1	5uL	1mL	0.05mg/L
Standard 2	10uL	1mL	0.10mg/L
Standard 3	25uL	1mL	0.25mg/L
Standard 4 (CCV)	50uL	1mL	0.50mg/L
Standard 5	75uL	1mL	0.75mg/L
Standard 6	100uL	1mL	1.0mg/L

9.2.3.3. Intermediate PCB ICV Standard Preparation

Dilute 100uL of the stock Aroclor 1016 ICV standard (100ug/mL), 100uL of the stock Aroclor 1260 ICV standard (100ug/mL) and 50uL of the Stock TCMX surrogate standard (200ug/mL) to 1mL with hexane to produce a final concentration of 10ug/mL for each component. Intermediate PCB ICV Standards of other Aroclors can be prepared using this formula to facilitate the preparation of an ICV for an individual Aroclor other than 1016 or 1260**.**

9.2.3.4. Working PCB ICV Standard Preparation

Dilute 50uL of the Intermediate PCB ICV standard (10ug/mL) to 1mL with hexane to produce a final concentration of 0.5ug/mL.

9.2.3.5. Working TCMX Surrogate Standard

Dilute 50uL of the Stock TCMX Surrogate (200ug/mL) to 1mL with hexane to produce a final concentration of 10ug/mL.

9.2.3.6. Intermediate Single Point PCB Calibration Standard Preparation

The following are examples of intermediate calibration standards and could vary based on requirements:

Standard	Stock Standard amount	Final Volume in Hexane	Final PCB Concentration
Intermediate 1221	10uL	1mL	10ug/L
Intermediate 1232	10uL	1mL	10ug/L
Intermediate 1242	100uL	1mL	10ug/L
Intermediate 1248	100uL	1mL	10ug/L
Intermediate 1254	100uL	1mL	10ug/L

9.2.3.7. Working Single Point PCB Calibration Standard Preparation

The following are examples of working calibration standards and could vary based on requirements:

Standard	Stock Standard amount	Working TCMX Surr. Amount	Final Volume in Hexane	Final PCB/Surr Concentration
Working 1221	100uL	50uL	1mL	1ug/L/0.5ug/L
Working 1232	50uL	50uL	1mL	0.5ug/L
Working 1242	50uL	50uL	1mL	0.5ug/L
Working 1248	50uL	50uL	1mL	0.5ug/L
Working 1254	50uL	50uL	1mL	0.5ug/L

10. Calibration

10.1. Before the initial calibration standards are injected, it is advisable to perform routine injection port and column maintenance due to the sensitivity of the ECD detector.

10.2. **Initial Calibration**: Initial calibration standards are introduced into the GC from the lowest to highest concentration by direct injection. Five calibration points, at a minimum, are analyzed to evaluate linearity. The lowest calibration standard must be at or below the required reporting limit. Method 608 requires three calibration points, at a minimum. Refer to the Quality Manual for more information regarding calibration curves.

10.2.1 A mixture of Aroclor 1016 and 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. An initial 5-point calibration is performed using the mixture of Aroclors 1016 and 1260.

10.2.2 Standards of the other Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been used to describe the detector response. The standards for these other Aroclors should

be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the Aroclor 1016/1260 initial calibration curve.

10.2.3 In situations where only one or a few Aroclors are of interest for a specific project, the analyst may employ a five-point initial calibration of each of the Aroclors of interest and not use the 1016/1260 mixture or the pattern recognition standards.

10.2.4 For Method 608, if a client sample contains an Aroclors other than 1016/1260, quantitation must be performed using a calibration curve for the appropriate Aroclor.

10.3. Record the peak area (or height) for each Aroclor peak to be used for quantitation. A minimum of 3 peaks must be chosen for each Aroclor, preferably 5 peaks are used. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 5 peaks should include at least one peak that is unique to that Aroclor. Use at least 5 peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors.

10.4. Calculate the Calibration Factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards using the calculation below:

$$CF = \frac{\text{Peak Area or Height in the Standard}}{\text{Mass of the Standard Injected, ng}}$$

Five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the 5 peaks chosen for this mixture. The single standard for each of the other Aroclors will generate at least 3 calibration factors, one for each selected peak.

10.5. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{CF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
CF_{avg} = Mean of CFs for a peak

10.6. If the %RSD of the CFs is $\leq$ 20% over the calibration range, then the slopes of the lines for each standard are sufficiently close to one another and the average CF may be used to determine sample concentrations. For Method 608, if the calibration factor is constant, < 10% RSD over the working range, linearity through the origin can be assumed and the average calibration factor can be used in place of a calibration curve.

10.7. If any %RSD is >20%, or >10% for Method 608, the analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.8 The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$r = \frac{\sum_{i=1}^{N} (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} (Y_i - \overline{Y})^2\right)}}$$

10.9 Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be ≥ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$\text{COD} = \frac{\sum_{i=1}^{N} (Y_i - \overline{Y})^2 - (\frac{n-1}{n-p}) \sum_{i=1}^{N} (Y_i - Y_i')^2}{\ldots\ldots\ldots\ldots \sum_{i=1}^{N} (Y_i - \overline{Y})^2}$$

10.10 When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the calibration model chosen for this mixture must be applied to the other 5 Aroclors for which only single standards are analyzed. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate linearity.

10.11 Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.12 Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy of the standards, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent difference (%D) of the observed ICV response from the initial calibration average response, according to the following equation where CF_v is the calibration factor of the verification standard:

$$\% \text{ Difference } (\%D) = [CF_{avg} - CF_v] / CF_{avg} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. All compounds must have a

maximum percent difference of +/- 30% from the initial calibration curve. **BP requires the ICV to be +/-15% D, based on true concentration.**

10.13 ICV Corrective Action: If the ICV fails the criteria, another ICV may be analyzed. If the second ICV fails, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV fails during an overnight run and is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

10.14 Daily and Continuing Calibration: Verify the initial calibration each 12-hour shift by injecting a Continuing Calibration Verification (CCV) standard prior to conducting any sample analyses.

The CCV must also be analyzed at intervals of not less than once every 20 samples and at the end of the analysis sequence. **BP requires that a CCV be analyzed every 10 samples.** For Aroclor analyses, the CCV standard should be a mixture of Aroclors 1016 and 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition.

10.15 The calibration factor (CF_v) for each analyte calculated from the CCV must not exceed a %Difference (%D) of more than +/-15% when compared to the CF_{avg} from the initial calibration curve.

10.16 CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11 Procedures

11.1 All sample extracts must be analyzed at room temperature and the system must be tuned and calibrated as per Section 10, and free of contamination before samples are analyzed.

11.2 Gas Chromatography conditions: Configure the GC per manufacturer's instructions.

11.3 Inject 1uL aliquots of all samples and quality control samples into the GC under the same operating conditions as used for the calibration standards. The sample vials are loaded onto the autosampler that is programmed via the data system to inject the necessary volume.

11.4 Qualitative Analysis

11.4.1 Absolute retention times are used for the identification of PCBs as Aroclors. Retention time windows are established on both the primary and the confirmation column to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. To establish retention time windows, make three or more injections of a standard over the course of a 72-hour period, at a minimum. Calculate the mean and standard deviation of the absolute retention times of the standard. The retention time window is defined as +/-3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater.

11.4.2 Tentative identification of a PCB compound is made when peaks from the sample extract fall within the established retention time windows for Aroclor peaks. Tentative Aroclor identification must be confirmed using a second GC column of dissimilar stationary phase, based on a clearly

identifiable Aroclor pattern, or using another technique such as GC/MS. Qualitative confirmation of Aroclors through separate injection onto a dissimilar column does not require the confirmation column to meet the calibration criteria outlined in Section 10.

11.5 Quantitative Analysis

11.5.1. The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

11.5.2 Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen and the calibration model established from the initial calibration curve. A concentration is determined using each of the characteristic peaks and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor.

11.5.3 When PCB concentrations are above the calibration range, the sample extract should be rerun at a dilution.

11.6 When the average calibration factor (CF_{avg}) from the initial calibration is used, calculate the on-column amount:

$$X_s = A_s/CF_{avg}$$

Where: X_s = On-column concentration of the analyte or surrogate
A_s = Total area or response of the analyte or surrogate
CF_{avg} = Average calibration factor from the initial calibration

11.7 When a linear curve fit is used, calculate the on-column amount:

$$X_s = (y - b)/a$$

Where: X_s = On-column concentration of the analyte or surrogate
y = Total area or response of the analyte or surrogate
a = slope of the line (the coefficient of x)
b = intercept of the line

11.8 When a non-linear or quadratic curve fit is used, calculate the on-column amount:

$$X_s = \frac{-b + \sqrt{b^2 - 4a(c - A_s)}}{2a}$$

Where: X_s = On-column concentration of the analyte or surrogate
A_s = Peak area or height of the analyte or surrogate in the sample
a = Slope of the line
b = The intercept
c = coefficient of the polynomial

11.9 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_t)(D)}{(V_i)(V_s)} \qquad \text{Solid Sample (ug/Kg)} = \frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$$

Where: X_s = On-column concentration of the analyte in the sample aliquot injected
V_t = Total volume of concentrated extract, in microliters
D = Dilution factor
V_i = Volume of the extract injected, in microliters
V_s = Volume of aqueous sample extracted in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$$

12 Quality Control

12.1 Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water or sodium sulfate	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 2) If a contaminant is present only in the method blank and not the samples, the sample data may be reported without qualifiers, the method blank must be qualified.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample results are non-detect, the sample results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

12.2 LCS equation

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD calculations:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13 Method Performance

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

13.2 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study.

14 Pollution Prevention and Waste Management

14.1 Procedures for handling waste generated during this analysis are addressed in S-ALL-S-002, Waste Handling or other applicable SOP.

14.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15 References

15.1 "Test Methods for Evaluating Solid Wastes", EPA SW-846, methods 8082, 8000.

15.2 40CFR, Part 136, Appendix A, EPA Method 608.

15.3 Pace Analytical Quality Manual; latest revision.

15.4 TNI Standard; Quality Systems section; latest revision.

16 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

Not Applicable

17 Revisions

Document Number	Reason for Change	Date
S-IN-O-050-rev.8	1. Added new section 10.2 regarding when and how to drop points from an initial calibration curve (copied from Quality Manual). 2. Section 3.3: revised soil reporting limit. 3. Table 7.1: added hold times for wipes and oils. 4. Table 8.1: revised column information to match current practices. 5. Section 9.2.3.2: indicated which standard is the CCV. 6. Section 10.8: added statement about 72-hour window for retention time studies. 7. Section 10.9: added last line about rerunning samples. 8. Section 10.11: made some wording changes for clarity (12 hour clock, CCVs every 20 per method, etc.) 9. Section 11.1: revised temperature program information. 10. Table 12.1: revised LCS and MS limits. 11. Section 15.1: revised method references. 12. Section 9.2.3.3: added surrogate to the ICV mix. 13. Section 9.2.3.5: added surrogate to the single point PCB standards. 14. Section 10.4: indicated that single point Aroclors (besides 1016 and 1260) must be run with each new initial calibration. 15. Section 16: added method reference of 8082A to allow use of TCMX as a surrogate. 16. Added new section 12.4 concerning dilutions and reruns. 17. Section 4.2: added in both cleanup methods available. 18. Added section 10.12 regarding the inability to use Grand Mean Averaging. 19. General: revised SOP references where applicable. 20. Section 10.9: added language for BP and Marathon requirement for ICV limits of 85-115%. 21. Removed old sections 11.7 thru 11.9 per SRS.	12Mar2008
S-IN-O-050-rev.9	1. Revised cover page to new format. 2. Updated Table of Contents to include Sections 10 and 11 3. Section 2.3: changed 2uL injection volume to 1uL 4. Section 3.3: added reporting limits for wipes and oils 5. Table 7.1: changed wipes and oils holding time for extraction to 14 days. 6. Section 8: added language to include the use of equivalent supplies. 7. Table 9.3: Updated standard used for 1221 and 1232 stock and added language to include the use of equivalent standards and changed PCB to TCMX for Surrogate Standard. 8. Section 9.2.3: added instructions for the preparation of the Working TCMX Surrogate and updated preparation instructions for single point standards. 9. Section 10.2: changed 2uL injection volume to 1uL. 10. Section 10.7: corrected linear and non-linear curve fit information and removed the incorrect regression equation. 11. Section 11.1: updated GC settings 12. Section 11.2: changed 2uL injection volume to 1uL. 13. Section 16: removed Tables 1 and 2	21Sep2009

S-IN-O-050-rev.10	1. Updated method reference to include EPA 608. 2. Section 1: added 608 reference. 3. Section 3.3: added that RLs may vary 4. Table 9.3: added that other Aroclors may be used in ICALs/ICVs 5. Section 9.2.3.1: added that other Aroclors may be used 6. Section 9.2.3.3: added that other Aroclors may be used 7. Section 10.2: added 608 requirement of 3 point curve at a minimum 8. Section 10.4: added 608 requirement of curve for quantitation 9. Section 10.6: added calculation for CF and replaced RF with CF in RSD and SD calculation. 10. Section 10.7: added calibration requirements for 608. 11. Section 10.11: added +/-15%D criteria for CCV and added %D calculation. Clarified corrective action steps. 12. Section 14.1: added "or other appropriate SOP". 13. Section 15: added EPA Method 608 reference	25Mar2010
S-IN-O-050-rev.11	1. Section 3: added Aroclors 1262 and 1268 to target list. Added language that limits, volumes, vendors, etc. are subject to change. 2. Table 8.1: updated with current information 3. Table 9.2: changed "second source" to ICV. For spiking standard, changed all target analytes to method specified analytes. 4. Table 9.3: updated with current information and added single point intermediate and working standards. 5. Section 9.2.3: updated with current practice and changed lists of standards to tables. 6. Section 10: completely reorganized and expanded to include equations for all curve fits and corrective actions for each calibration step. 7. Section 11: reorganized and expanded to include equations for determining on-column concentrations for each curve fit and moisture corrected concentration. 8. Table 12.1: updated and added reference to LIMS for control limits. 9. Section 13.1: removed SOP reference. 10. Section 15.4: changed NELAC to TNI	11Apr2012

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STANDARD OPERATING PROCEDURE

SEPARATORY FUNNEL EXTRACTION
REFERENCE METHOD: EPA SW-846 METHOD 3510C

SOP NUMBER:	S-IN-O-054-rev.12
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-O-054-rev.11

APPROVAL

Karl Anderson
General Manager — April 18, 2013 Date

Beth Schrage
Quality Manager — April 16, 2013 Date

Steven R Long
Department Manager — April 16, 2013 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for extracting non-volatile and semi-volatile organic compounds from groundwater and surface water samples in a separatory funnel while meeting the requirements specified in EPA method 3510C.

2. Summary of Method

2.1. A measured volume of sample, normally about 1 liter, is serially extracted with solvent in a separatory funnel. Reduced sample volumes may be used providing that the ratio of sample to solvent remains consistent with the ratio indicated in Method 3510C. Reduced sample volume extraction may also be referred to as Low Volume Extraction (LVE). Some extractions also require the monitoring and adjusting of the pH of the sample. The extract is separated from the sample and is concentrated, followed by cleanup, if necessary, or analysis.

3. Scope and Application

3.1. This procedure is for extracting water insoluble or slightly water soluble organic compounds from groundwater, surface water and other aqueous samples using methylene chloride as the extraction solvent.

3.2. This procedure is applicable for the extraction of semi-volatile compounds including BNAs, PAHs, PCBs, and Diesel Range Organics/Extended Range Organics (DRO/ERO).

3.3. Applicable compounds, volumes/weights utilized, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.4. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of separatory funnel equipment and reagents. Each analyst work cell must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Solvents, reagents and glassware can all contribute to compound artifacts or raised baselines; both conditions that can affect chromatography. Analyzing method blanks is therefore crucial in determining the presence of contaminants.

4.2. Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

5.2. Samples

Take precautions when handling samples. Samples must always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L or 125mL widemouth, or equivalent.	None	0°C to 6°C	Samples must be extracted within 7 days of collection date and must be analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment/Instrumentation

Equipment	Vendor	Description / Comments
N-EVAP concentrator	Organomation	Or equivalent equipment
Zymark concentrator (2) and glassware	Zymark	Or equivalent equipment
Shaker Tables	Glas-Col	Or equivalent equipment

8.2. General Supplies

Item	Description
Separatory Funnels	2L or 125mL, Teflon, with PTFE stopcocks and Teflon lids or equivalent
Glass beakers	400mL Pyrex or equivalent
Autosampler vials	~2mL, clear glass with aluminum crimp-top seals
Micro-syringes	Various sizes
Glass funnels	
Glass wool	
Graduated cylinders	Glass, Class A
Kuderna-Danish Concentrator Sets	500mL flash with 10mL concentrator tube and 3-ball Snyder column
Heated water bath	Temperature controlled
Boiling Chips	Teflon or equivalent
Pasteur pipettes	For dispensing acid or base
pH paper	pH range 1-12
Glass stirring rods	For breaking up emulsions
Glass tubes	Disposable, 20x150mm or equivalent
Filter paper	For filtration of extract
Pipettes	Volumetric, Class A, various sizes

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Reagent water	De-ionized water, ASTM Type II water
Sodium Sulfate	Anhydrous, granular 10-60 mesh, meets ACS specs or equivalent
Methylene Chloride	Extraction solvent, pesticide grade or better
Acetone	Extraction solvent, pesticide grade or better
Hexane	Exchange solvent, pesticide grade or better
Sulfuric acid solution	1:1 solution
Sodium Hydroxide solution (10N)	Dissolve 400g sodium hydroxide pellets into 1L of reagent water or purchase premade

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogates are added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains all target analytes.	Same solution can be used for the LCS and MS/MSD

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock BNA/BNA LVE spike standard	1. Restek; catalog # 31004, B/N spike; 1000ug/mL, or equivalent 2. Restek; catalog # 31014, Acid spike; 2000ug/mL, or equivalent 3. Restek; catalog # 561763, Custom PAH spike; 5000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions.
Working BNA/BNA LVE spike standards	Refer to Section 9.2.3.1	Good for 6 months from preparation date	Same as stock standard.
Stock BNA/BNA LVE surrogate standard	O2si; catalog # 11004-83-1L; 100mg/L, or equivalent. Use 1mL for each BNA. Use 100uL for each BNA LVE.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions.
Stock/Working Full-list BNA/Full list BNA LVE spike standard	NSI Solutions; catalog #WL-409; TCL BNA Spike; 100ug/mL or equivalent. Use 1mL for each Full list BNA LCS, MS and MSD. Use 100uL for each Full list BNA LVE LCS, MS and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions.

Standard Type	Description	Expiration	Storage
Stock PCB/PCB LVE spike standard	Restek; catalog # 32029, Aroclor 1016/1260; 1000ug/mL in hexane, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PCB/PCB LVE spike standard	Refer to Section 9.2.3.2	Good for 6 months from preparation date	Same as stock standard.
Stock PCB/PCB LVE surrogate standard	1. Restek; catalog # 32028, 2,4,5,6-tetrachloro-m-xylene (TCMX); 200ug/mL in acetone, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PCB/PCB LVE surrogate standard	Refer to Section 9.2.3.3	Good for 6 months from preparation date	Same as stock standard.
Stock DRO/DRO LVE spike standard	Restek; catalog # 31258, Diesel #2 standard; 50,000ug/mL in methylene chloride, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working DRO/DRO LVE spike standard	Refer to Section 9.2.3.4 and Section 9.2.3.5	Good for 6 months from preparation date	Same as stock standard.
Stock DRO/DRO LVE surrogate standard	Restek; catalog # 31487, Pentacosane; 10,000ug/mL in methylene chloride, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working DRO/DRO LVE surrogate standard	Refer to Section 9.2.3.6 and Section 9.2.3.7	Good for 6 months from preparation date	Same as stock standard.
Stock PAH-SIM/PAH-SIM LVE spike standard	Restek; catalog # 31622, Cal. Mix 5; 2000ug/mL of each compound in methylene chloride, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PAH-SIM/PAH-SIM LVE spike standard	Refer to Section 9.2.3.8	Good for 6 months from preparation date	Same as stock standard.
Stock PAH-SIM/PAH-SIM LVE surrogate standard	Restek; catalog # 31062, B/N surrogate; 5000ug/mL of each compound in methylene chloride, or equivlent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PAH-SIM/PAH-SIM LVE surrogate standard	Refer to Section 9.2.3.9	Good for 6 months from preparation date	Same as stock standard.
Stock/Working Scan/SIM Combo spike standard	O2si, catalog #114072-06, 10-100ug/mL or equivalent. Use 1ml for each LCS, MS and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Stock/Working Scan/SIM Combo and Scan/SIM Combo LVE surrogate spike standard	O2si, catalog #114071-06; 10-100ug/mL or equivalent. Use 1mL for each sample, Method Blank, LCS, MS and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Stock TCLP BNA spike standard	1. Restek; catalog # 31028, TCLP B/N spike; 2000ug/mL of each compound in acetone or equivalent 2. Restek; catalog # 31027, TCLP Acid spike; 2000ug/mL of each compound in methanol or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working TCLP BNA spike standard	Refer to Section 9.2.3.10	Good for 6 months from preparation date	Same as stock standard.

9.2.3. Standard Preparation Procedures

9.2.3.1. Working BNA/BNA LVE Spike Standard Preparation

Dilute 2.5mL of the stock Acid spike standard (2000ug/mL), 5mL of the stock B/N spike standard (1000ug/mL) and 1mL of the stock Custom PAH spike (5000ug/mL) to 50mL with acetone for a final concentration of 100ug/mL. Add 1mL of this working standard to each BNA LCS, MS and MSD. Add 100uL of this working standard to each BNA LVE LCS, MS and MSD.

9.2.3.2. Working PCB/PCB LVE Spike Standard Preparation

Dilute 1mL the stock PCB/PCB LVE spike standard, Aroclors 1016/1260, (1000ug/mL each) to 200mL with acetone for a final concentration of 5ug/mL. Add 1mL of this working spike to each PCB LCS, MS and MSD. Add 50uL of this working spike to each PCB LVE LCS, MS and MSD.

9.2.3.3. Working PCB/PCB LVE Surrogate Standard Preparation

Dilute 5mL of the stock PCB/PCB LVE surrogate standard, TCMX (200ug/mL) to 500mL with acetone for a final concentration of 2ug/mL. Add 1mL of this working surrogate to each PCB sample, Method Blank, LCS, MS and MSD. Add 50uL of this working surrogate to each PCB LVE sample, Method Blank, LCS, MS and MSD.

9.2.3.4. Working DRO Spike Standard Preparation

Dilute 12.5mL of the stock DRO/DRO LVE spike standard (50,000ug/mL) to 250mL with acetone for a final concentration of 2500ug/mL. Add 1mL of this working standard to each DRO/ERO/Ohio mod LCS, MS and MSD.

9.2.3.5. Working DRO LVE Spike Standard Preparation

Dilute 12.5mL of the stock DRO/DRO LVE spike standard (50,000ug/mL) to 25mL with acetone for a final concentration of 25,000ug/mL. Add 100uL of this working standard to each DRO/ERO/Ohio mod LVE LCS, MS and MSD.

9.2.3.6. Working DRO Surrogate Standard Preparation

Dilute 7.5mL of the stock DRO/DRO LVE surrogate standard (10,000ug/mL) to 500mL with acetone for a final concentration of 150ug/mL. Add 1mL of this working surrogate to each DRO/ERO/Ohio mod sample, Method Blank, LCS, MS and MSD.

9.2.3.7. Working DRO LVE Surrogate Standard Preparation

Dilute 7.5mL of the stock DRO/DRO LVE surrogate standard (10,000ug/mL) to 50mL with acetone for a final concentration of 1,500ug/mL. Add 100uL of this working surrogate to each DRO/ERO/Ohio mod LVE sample, Method Blank, LCS, MS and MSD.

9.2.3.8. Working PAH-SIM/PAH-SIM LVE Spike Standard Preparation

Dilute 5mL of the stock PAH-SIM/PAH-SIM LVE spike standard (2000ug/mL) to 200mL with acetone for a final concentration of 50ug/mL. Add 200uL of this working spike to each PAH-SIM LCS, MS and MSD. Add 20uL of this working spike to each PAH-SIM LVE LCS, MS and MSD.

9.2.3.9. Working PAH-SIM/PAH-SIM LVE Surrogate Standard Preparation

Dilute 5.0mL of the stock PAH-SIM/PAH-SIM LVE surrogate standard (5000ug/mL) to 500mL with acetone for a final concentration of 50ug/mL. Add 200uL of this working surrogate to each PAH-SIM sample, Method Blank, LCS, MS and MSD. Add 20uL of this working surrogate to each PAH-SIM LVE sample, Method Blank, LCS, MS and MSD.

9.2.3.10. Working TCLP BNA Spike Standard Preparation

Dilute 1mL of the stock TCLP acid spike standard (2000ug/mL) and 1mL of the stock TCLP B/N spike standard (2000ug/mL) to 20mL with acetone for a final concentration of 100ug/mL. Add 1mL of this working spike to each TCLP BNA LCS, MS and MSD.

10. Calibration

10.1. Not applicable to this SOP.

11. Procedures

11.1. Make sure that all glassware and Teflon separatory funnels used for this procedure have been properly washed. All washed glassware must be rinsed prior to use with acetone to remove residual water and with methylene chloride to remove any residual contaminants.

11.2. Measure the initial pH of each sample using wide range pH paper. Dip a clean disposable Pasteur pipette into each sample and touch the pipette to a piece of pH paper. Record the initial pH in the extraction log.

11.3. A nominal volume of 1L or 100mL of aqueous sample is normally extracted. For samples expected to contain high concentrations of analytes, use a smaller aliquot of sample diluted to 1L or 100mL with reagent water.

11.4. For each batch of 20 or fewer samples, prepare a method blank and an LCS by using aliquots of reagent water. The method blank will be used to check for contamination in the system. The LCS will be used to determine the efficiency of the extraction method in extracting target compounds. MS/MSD should also be prepared if sample volume allows.

11.5. Pour the aqueous sample into a clean separatory funnel and place the funnel in a ring stand on a secured rack. Alternatively, measure the desired sample volume into a clean graduated cylinder and transfer to a clean separatory funnel on a secured rack. Water samples with excessive amounts of loose sediment in the jar should have the water portion carefully decanted into the separatory funnel or graduated cylinder in order to minimize the amount of sediment that is transferred.

11.6. Add the appropriate **surrogate** solution to each method blank, sample, LCS and MS/MSD. Add the appropriate **spiking** solution to the LCS and the MS/MSD. Refer to the standard preparation log and the sample preparation log for details regarding the appropriate spiking solution and volume to be used.

11.7. Adjust the sample pH, if necessary, to the pH indicated in Table 1 using 1:1 Sulfuric Acid or 10N Sodium Hydroxide. The pH is checked by dipping the tip of a disposable glass pipet into each well-mixed sample and placing the tip onto the pH paper to obtain a pH measurement.

11.8. Rinse the 1L sample jar with a 60mL portion of extraction solvent or rinse a 100mL sample jar with a 6mL portion of extraction solvent, see Table 1, and transfer the rinsate to the separatory funnel. If the entire contents of the sample jar is not used and a sample aliquot is transferred using a graduated cylinder, rinse the graduated cylinder with the solvent portion and transfer the rinsate to the separatory

funnel. If the sample jar contains a significant amount of loose sediment after decanting the water portion, the sediment should be removed and discarded before the jar is rinsed with solvent and the solvent portion is transferred to the separatory funnel. Samples with excessive sediment should be noted in the extraction log.

11.9. Seal the separatory funnels with Teflon lids and shake for two minutes with periodic venting. This can be done manually or on an automatic shaker. **NOTE**: Methylene chloride may cause excessive pressure in the separatory funnel. It is recommended to shake slightly and vent before placing funnels on an automatic shaker.

11.10. Return the 2L separatory funnels to their ring stands and allow the solvent layer to separate from the aqueous phase. The 125mL separatory funnels remain in the automatic shaker for draining. If an excessive emulsion is present in the solvent layer, it can be broken up manually by using a clean glass stirring rod. If this is not successful, the sample can be drained into a clean secondary container (i.e. VOA vial) and transferred to a centrifuge tube. The extract can be centrifuged and then decanted into the drying funnel.

11.11. Drain the solvent layer through a drying funnel consisting of a clean funnel containing a plug of clean glass wool topped with a portion of clean sodium sulfate. The solvent should be collected in labeled beakers, labeled KD glassware, or labeled glass tubes.

11.12. Repeat the extraction two additional times with 60mL or 6mL aliquots of solvent. BNA LVE extractions require only one additional extraction – see Table 1. Drain the solvent through the drying funnel after each extraction. After the final solvent extraction has been collected, rinse the funnel with methylene chloride and remove the drying funnel.

11.13. If extraction at a secondary pH is required, add acid or base as necessary and serially extract the sample, as described in Sections 11.9-11.12, at the adjusted pH. Collect all sample extract fractions together for concentration. Refer to Table 1 for extraction conditions.

11.14. Once extraction is complete, initial sample volume may be determined, by pouring the sample from the separatory funnel into a Class A, glass graduated cylinder and recording the sample volume in mLs.

11.15. **Concentration procedure for 8270 BNA or Scan/SIM Combo samples:** Pour the extract into a labeled Kuderna-Danish concentrator with a concentrator tube securely attached. Add one or two clean boiling chips to the KD flask and attach a 3-ball Snyder column. Place the KD apparatus on a hot water bath so that the flask is partially immersed in the water. At the proper rate of distillation, the balls of the Snyder column should actively chatter but the chambers should not flood with solvent. Adjustment of the angle of the apparatus and the water temperature may be necessary to make the boiling more efficient. When the apparent volume of the extract reaches 4-6mL, remove the apparatus from the water bath and allow it to cool. Once cooled, carefully disassemble the KD apparatus rinsing each joint into the concentrator tube with a small amount of extraction solvent. Place the concentrator tube into the N-Evap and further concentrate the extract until the apparent volume is slightly below 1mL. Continue to Section 11.19.

11.16. **Concentration of LVE samples:** Pour the extract into a labeled 20x150mm glass tube and place the tube on the N-Evap concentrator in a warm water bath (about 40^{o}C) and evaporate the solvent volume using a gentle stream of nitrogen. During the process, the tube should be rinsed several times with the appropriate solvent. The tube should be positioned so that water will not condense into the sample and the sample should not be allowed to go below 0.5mL, this could lead to losses of semi-volatile compounds.

11.17. **Concentration procedure for all other samples:** Pour the entire sample extract into a labeled Zymark extractor tube and place in the Zymark concentrator apparatus. Adjust the settings per manufacturer's instructions. When the apparent volume is slightly below the intended final volume,

remove the concentrator from the apparatus and allow it to cool.

11.18. If a solvent exchange is required, see Table 1, add 50mL of the exchange solvent to the Zymark tube. Concentrate the extract to slightly below the intended final volume, remove from the water bath and allow it to cool.

11.19. If further concentration is necessary for any sample extract, nitrogen blowdown can be performed. For this procedure, place the concentrator tube on the N-Evap concentrator in a warm water bath (about 40^{o}C) and evaporate the solvent volume using a gentle stream of nitrogen. During the process, the tube should be rinsed several times with the appropriate solvent. The tube should be positioned so that water will not condense into the sample and the sample should not be allowed to go below 0.5mL, this could lead to the loss of semi-volatile compounds.

11.20. Prepare a calibrated vial by volumetrically dispensing the required volume of the solvent being used into a vial and securely capping the vial to eliminate evaporation. The calibrated vial must be prepared daily. Quantitatively transfer the sample extract from the Zymark tube or concentrator tube to a vial. Bring the sample extract in the vial to the required final volume listed in Table 1 by visually comparing the sample extract vial volume to the calibrated vial volume. Securely cap the sample extract vial. Store all extracts in the appropriate storage cooler. For extracts that will not concentrate to the usual final volume, use the procedure described above to bring the extract to the next higher practical volume for which a calibrated vial can be prepared.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, affected data must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to LIMS for control limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to LIMS for control limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to LIMS for control limits.	Samples with surrogate failures must be re-extracted and reanalyzed. QC samples with surrogate failures require the re-extraction and reanalysis of the QC samples and the associated client samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) If only one surrogate for BNA or Combo fails and it is >10% recovery, re-extraction is not required but affected data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

12.2. LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

12.3. MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13. Method Performance

13.1. Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

13.2. Demonstration of Capability (DOC): Analysts performing this method must document acceptable accuracy and precision by passing a demonstration of capability study (DOC) on an annual basis.

14. Method Modifications

14.1. Spikes not added to graduated cylinder or sample bottle but instead added to the separatory funnel.

14.2. BNA LVE performed with two solvent portion extractions at each pH instead of three solvent portion extractions.

14.3. Initial sample volume is determined by directly measuring the sample volume in the separatory funnel after the extraction is complete instead of marking the sample container and filling the container to the mark with water.

15. Pollution Prevention and Waste Management

15.1. Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling or other applicable SOP.

15.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

15.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16. References

16.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3510 "Separatory Funnel Extraction".

16.2. Pace Analytical Quality Manual; latest revision.

16.3. TNI Standard; Quality Systems section; latest revision.

17. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1. Table 1: Extraction Conditions

18. Revisions

Document Number	Reason for Change	Date
S-IN-O-054-rev.8	1. Updated cover page format 2. Section 8: added 40mL vials to supplies list 3. Section 9: updated lists to accommodate equivalent brands 4. Section 9: removed references to pesticides and TN-EPH 5. Section 11.3: inserted initial pH determination procedure 6. Sections 11.8.1, 11.8.2 and 11.8.3: removed and included in Table 1 7. Section 11.14: added 8. Section 11.20: removed references to soils, pesticides and EPH 9. Table 1: added	10Sep2009
S-IN-O-054-rev.09	1. Section 3: removed reference to pesticides. Added language that volumes/weights, vendors, etc… are subject to change. 2. Section 4: removed reference to extract cleanup procedures. 3. Table 7.1: updated/clarified storage and hold time information. 4. Section 8: updated information and added shaker tables to equipment list. 5. Table 9.3: updated standards and added full-list BNA standard and Scan/SIM Combo standard. 6. Section 11: added references to BNA Scan/SIM Combo samples. Added detail regarding the use of KD setups. Updated final extract determination technique to the comparison vial method. 7. Table 12.1: updated and clarified exceptions. Referred to LIMS for control limits. 8. Section 15: changed NELAC to TNI. 9. Table 1: updated to include BNA Scan/SIM Combo and TCLP BNA.	03May2011
S-IN-O-054-rev.10	1. Section 2.1: added language for use of reduced sample volumes or LVE. 2. Section 3.2: added ERO 3. Table 7.1: added sample container for reduced sample volume extraction or LVE 4. Table 8.2: added equipment for reduced volume extraction or LVE 5. Sections 9.2.3.7 and 9.2.3.8: added spiking instructions for LVE. 6. Section 11.3: added language for reduced volume extraction or LVE 7. Section 11.6: moved previous Section 11.7 to Section 11.6 to reflect actual practice. 8. Section 11.7: added language for reduced volume extraction or LVE 9. Section 11.10: added language for reduced volume extraction or LVE 10. Section 11.11: replaced "1-2 inches" with "a portion" because the funnels for the reduced volume samples won't hold 1-2 inches of sodium sulfate. 11. Section 11.12:added language for reduced volume extraction or LVE 12. Section 11.16: added for reduced volume extraction or LVE 13. Section 11.19: specified equipment as N-Evap concentrator 14. Section 14.1: revised SOP reference.	20Mar2012

S-IN-O-054-rev.11	1. Previous Section 11.7 becomes Section 11.8. 2. Section 11.7 expanded to clarify the pH checking and adjustment process. 3. Inserted new Method Modifications section.	12Sep2012
S-IN-O-054-rev.12	1. Table 7.1: added "or equivalent" for sample bottle type 2. Table 8.2: added volumetric pipettes 3. Table 9.3: added detail for standards used for LVE. 4. Section 9.2.3: added instruction for preparation and use of standards for LVE. 5. Section 11.5: added option to measure sample volume prior to transfer to separatory funnel. 6. Section 11.12: added BNA LVE requirement for two total extractions per fraction and added reference to Table 1. 7. Section 11.13: added reference to previous sections for instructions. 8. Section 11.14: added language to describe option of determining sample volume if not already known. 9. Section 11.20: added instruction for determining non-standard final volumes. 10. Section 13.2: updated DOC requirements 11. Section 14.2: added BNA LVE modification of number of extractions and initial volume determination modification. 12. Table 1: added # of extractions per pH fraction information.	16Apr2013

Table 1 – Extraction Conditions

Determinative Method	Initial Extraction pH	# of extractions at Initial pH	Secondary Extraction pH	# of extractions at Secondary pH	Extraction Solvent	Exchange Solvent	Final Extract Volume (mL)
8015 DRO/DRO LVE (includes ERO and Ohio mod)	<2	3	N/A	N/A	Methylene Chloride	N/A	1
8082 PCB/PCB LVE	5-9	3	N/A	N/A	Methylene Chloride	Hexane	10
8270 PAH-SIM/PAH-SIM LVE	>11	3	N/A	N/A	Methylene Chloride	N/A	1
8270 BNA	<2	3	>11	3	Methylene Chloride	N/A	1
8270 BNA LVE	<2	2	>11	2	Methylene Chloride	N/A	1
8270 BNA Scan/SIM Combo	<2	3	>11	3	Methylene Chloride	N/A	1
8270 BNA Scan/SIM Combo LVE	<2	2	>11	2	Methylene Chloride	N/A	1
8270 TCLP BNA	<2	3	>11	3	Methylene Chloride	N/A	1

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SEMI-VOLATILE COMPOUNDS BY GC/MS

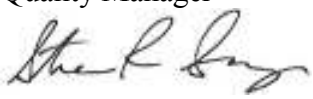
REFERENCE METHOD: EPA SW-846 METHOD 8270C

SOP NUMBER:	S-IN-O-068-rev.12
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-O-068-rev.11

APPROVAL

General Manager — May 8, 2013 — Date

Quality Manager — May 07, 2013 — Date



Department Manager — May 07, 2013 — Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of semi-volatile organic compounds in sample extracts while meeting the requirements specified in EPA method 8270C.

2. Summary of Method

2.1. Semi-volatile compounds are introduced into a gas chromatograph by injection of a sample extract onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes that are then detected with a mass spectrometer. Identification of the analytes is made by comparing their mass spectra with spectra of known standards. Quantitation is accomplished by comparing the response of a major ion relative to the internal standard response using a multi-point calibration curve.

2.2. Method 8270C provides chromatographic conditions for the detection of semi-volatile compounds in organic extracts. Aqueous samples are extracted using SW-846 method 3510 Separatory Funnel Extraction or other applicable method. Solid samples are extracted using SW-846 method 3546 Microwave Extraction or other applicable method.

3. Scope and Application

3.1. The list of compounds and reporting limits analyzed by this laboratory for method 8270C is found in Table 1. Other compounds may be reported upon completion of appropriate validation procedures. Refer to the LIMS for method detection limits.

3.2. This method can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, phenols, and nitrophenols.

3.3. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent extraction and exhibits poor chromatographic behavior. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosdiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3.4. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.5. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS equipment and the interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Glassware for the preparatory steps for this method should be thoroughly cleaned and rinsed. Soap products can leave phthalates on the glassware that may appear in the analytical data. Hits for phthalates should be closely scrutinized and continuous hits should warrant checking on the glassware cleaning procedure.

5. Safety

5.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. The use of gloves, lab coats and safety glasses is required. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock semi-volatile standards are toxic and should be handled with extreme care.

5.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L or 125mL widemouth, or equivalent.	None required	0°C to 6°C	Must be extracted within 7 days of collection date and analyzed within 40 days of extraction date
Solid	> 200 grams in 4oz or 8oz glass jar	None required	0°C to 6°C	Must be extracted within 14 days of collection date and analyzed within 40 days of extraction date

Samples and sample extracts must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

Sample extracts must be stored in the refrigerator or freezer until analysis is complete.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatographs	Hewlett Packard/Agilent	Lab uses models 6890 and 7890	Or equivalent system
Data Systems	Hewlett Packard/Agilent	Chemstation	Or equivalent system
Autosamplers	Hewlett Packard/Agilent		Or equivalent system
Mass Spectrometers	Hewlett Packard/Agilent	Lab uses models 5973 and 5975	Or equivalent system

8.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Restek	DB5MS	15m x 0.25mm or equivalent column

8.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials (clear vials)	Supelco or equivalent
Autosampler vials	1.8mL clear vials	Or equivalent

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	Pesticide grade or equivalent
Acetone	Pesticide grade or equivalent
Methanol	Pesticide grade or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Tuning Standard	Standard used to tune the mass spectrometer	DFTPP solution
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains method required spiking compounds, at a minimum, and is used for spiking MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock 8270/8270 LVE calibration standards	O2si; catalog #110920-01, 1000-2000ug/mL or equivalent O2si; catalog #110931-01, 2000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate #1 8270 calibration standards	Refer to Section 9.2.3.1	Solution good for 6 months from preparation	Same as stock standard.
Working 8270/Intermediate #2 8270 LVE calibration standards	Refer to Sections 9.2.3.2.	Same expiration date as Intermediate #1 8270 calibration standard	Same as stock standard.
Working 8270 LVE calibration standards	Refer to Sections 9.2.3.3.	Same expiration date as Intermediate #1 8270 LVE calibration standard	Same as stock standard.
Stock 8270 ICV standard with surrogates	NSI; catalog #WL-509, 200ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working 8270/Intermediate 8270 LVE ICV standard	Refer to Section 9.2.3.4.	Solution good for 6 months from preparation	Same as stock standard.
Working 8270 LVE ICV standard	Refer to Section 9.2.3.5.	Same expiration date as Intermediate 8270 LVE ICV standard	Same as stock standard.
Working 8270/Stock 8270 LVE internal standards	Restek; catalog # 31006; 4000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working 8270 LVE internal standards	Refer to Section 9.2.3.6.	Solution good for 6 months from preparation	Manufacturer's recommended storage conditions
Stock 8270/8270 LVE Surrogate standards	Accustandard; catalog # M-8270-SS; 4000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Stock DFTPP Tuning Standard	Restek; catalog # 31615; 1000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working DFTPP/Intermediate DFTPP LVE Tuning Standard	Refer to Section 9.2.3.7.	Solution good for 6 months from preparation	Same as stock standard
Working DFTPP LVE Tuning Standard	Refer to Section 9.2.3.8.	Same expiration date as Intermediate DFPP LVE Tuning standard	Same as stock standard

9.2.3. Standard Preparation Procedures

9.2.3.1 Intermediate #1 8270 Calibration Standard

Combine 1mL of each Stock 8270 Calibration Standard (1000-2000ug/mL) plus 500uL of the Stock 8270 Surrogate Standard (4000ug/mL) and dilute to 10mL with methylene chloride for a final concentration of 100-200ug/mL.

9.2.3.2 Working 8270 Calibration Standards/Intermediate #2 8270 LVE Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate #1 Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Stock Internal Standard amount added to Cal. Std.	Internal Standard Concentration
8270 Calibration Std 1	2.5uL	500uL	1ug/mL	5uL	40ug/mL
8270 Calibration Std 2	12.5uL	500uL	5ug/mL	5uL	40ug/mL
8270 Calibration Std 3	25uL	500uL	10ug/mL	5uL	40ug/mL
8270 Calibration Std 4	50uL	500uL	20ug/mL	5uL	40ug/mL
8270 Calibration Std 5	125uL	500uL	50ug/mL	5uL	40ug/mL
8270 Calibration Std 6	250uL	500uL	100ug/mL	5uL	40ug/mL
8270 Calibration Std 7	375uL	500uL	150ug/mL	5uL	40ug/mL
8270 Calibration Std 8	437.5uL	500uL	175ug/mL	5uL	40ug/mL
8270 Calibration Std 9	500uL	500uL	200ug/mL	5uL	40ug/mL

9.2.3.3 Working 8270 LVE Calibration Standards

Prepare using the Working 8270 Calibration Standards as Intermediate Standards.
The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate #2 Cal. Std. ID from Section 9.2.3.2	Intermediate Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Internal Standard Concentration
8270 LVE Cal. Std 1	8270 Calibration Std 1	50uL	500uL	0.1ug/mL	4ug/mL
8270 LVE Cal. Std 2	8270 Calibration Std 2	50uL	500uL	0.5ug/mL	4ug/mL
8270 LVE Cal. Std 3	8270 Calibration Std 3	50uL	500uL	1.0ug/mL	4ug/mL
8270 LVE Cal. Std 4	8270 Calibration Std 4	50uL	500uL	2.0ug/mL	4ug/mL
8270 LVE Cal. Std 5	8270 Calibration Std 5	50uL	500uL	5.0ug/mL	4ug/mL
8270 LVE Cal. Std 6	8270 Calibration Std 6	50uL	500uL	10ug/mL	4ug/mL
8270 LVE Cal. Std 7	8270 Calibration Std 7	50uL	500uL	15ug/mL	4ug/mL
8270 LVE Cal. Std 8	8270 Calibration Std 8	50uL	500uL	17.5ug/mL	4ug/mL
8270 LVE Cal. Std 9	8270 Calibration Std 9	50uL	500uL	20ug/mL	4ug/mL

9.2.3.4 Working 8270 ICV Standard/Intermediate 8270 LVE ICV Standard

Dilute 250uL of the Stock 8270 ICV Standard (200ug/mL) to 500uL with methylene chloride for a final concentration of 100ug/mL.

9.2.3.5 Working 8270 LVE ICV Standard

Dilute 50ul of the Intermediate 8270 LVE ICV Standard (100ug/mL) to 500uL with methylene chloride for a final concentration of 10ug/mL.

9.2.3.6 Working 8270 LVE Internal Standards

Dilute 500uL of the Stock 8270 LVE internal standards (4000ug/mL) to 5mL with methylene chloride for a final concentration of 400ug/mL.

9.2.3.7 Working DFTPP Tuning Standard/Intermediate DFTPP LVE Tuning Standard

Dilute 25uL of the Stock DTPP Tuning Standard (1000ug/mL) to 500uL with methylene chloride for a final DFTPP concentration of 50ng/uL.

9.2.3.8 Working DFTPP LVE Tuning Standard

Dilute 50uL of the Intermediate DTPP LVE Tuning Standard (50ug/mL) to 500uL with methylene chloride for a final DFTPP concentration of 5ng/uL.

10. Calibration

10.1. DFTPP Tune Verification: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned using a 50ng injection of DFTPP. Analysis must not begin until the tuning criteria are met. Use the DFTPP mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected. The 12-hour window during which standards and samples may be analyzed begins with the injection of DFTPP. All subsequent standards, samples MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

Mass (m/z)	Ion Abundance criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

If the DFTPP ratios do not meet the criteria, reanalyze the DFTPP tune. If the DFTPP still fails the criteria, autotune adjustment, instrument maintenance, and/or preparation of new standards must be considered.

The mass spectrum of DFTPP may be obtained by averaging three scans, the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using this approach and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak. Alternatively, the analyst may use other approaches suggested below:

1. A single scan within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.

2. An average of multiple scans within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.

10.2. Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

10.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

10.4. The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

10.5. The %RSD should be should be $\leq$15% for each target analyte. However, the %RSD for each individual Calibration Check Compound (CCC) must be $\leq$30%. If the RSD of any CCC is >30%, then the chromatographic system is too reactive and instrument maintenance or preparation of new standards may be necessary before attempting recalibration. The CCCs are:

Base/Neutral Fraction	**Acid Fraction**
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

10.6. System Performance Check Compounds (SPCCs) are checked for a minimum average response factor (RF_{avg}) to determine potential instability and/or degradation caused by deterioration of instrument conditions or standard material. The minimum RF_{avg} for the semivolatile SPCCs are as follows:

N-nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Instrument maintenance or preparation of new standards may be necessary. The SPCC criteria must be met for sample analysis to begin.

10.7. If the percent relative standard deviation (%RSD) of the RFs for a compound is ≤15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

10.8. If the % RSD for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.9 The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$r = \frac{\sum_{i=1}^{N} (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} (Y_i - \overline{Y})^2\right)}}$$

10.10 Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be ≥ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$\text{COD} = \frac{\sum_{i=1}^{N} (Y_i - \overline{Y})^2 - \left(\frac{n-1}{n-p}\right) \sum_{i=1}^{N} (Y_i - Y_i')^2}{\cdots\cdots\cdots\cdots \sum_{i=1}^{N} (Y_i - \overline{Y})^2}$$

10.11 Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument

maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.12 Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. The ICV recoveries are evaluated against a default acceptance range of 70-130% recovery.

10.13 ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

10.14 Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a DFTPP tune verification as described in Section 10.1, followed by a Continuing Calibration Verification (CCV) standard.

10.15 If the % difference (%D) or % Drift for each CCC is $\leq$20%, then the initial calibration is assumed to be valid. The response factors for all SPCCs in the CCV standard must meet the criteria in Section 10.6. **For BP, surrogate compounds must also be $\leq$20%D/Drift.**

10.16 The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

10.17 CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11 Procedures

11.1 All sample extracts must be analyzed at room temperature and the system must be tuned and calibrated as per Section 10, and free of contamination before samples are analyzed.

11.2 Gas Chromatography conditions: Configure the GC/MS per manufacturer's instructions.

11.3 The 1mL extract obtained from sample preparation for 8270 should be fortified with 10uL of the Stock 8270 internal standard (4000ug/mL) just prior to analysis such that 40ng of internal standard is injected onto the column. The 1mL extract obtained from sample preparation for 8270 LVE should be fortified with 10uL of the Working 8270 LVE internal standard (400ug/mL) just prior to analysis such that 4ng of

internal standard is injected onto the column. Analyze each 8270 extract by injecting 1uL onto the column. Analyze each 8270 LVE extract by injecting 10uL onto the column.

11.4 Qualitative Analysis

11.4.1 The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

11.4.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for characteristic ions.

11.4.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.5 Quantitative analysis: Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. See Sections 11.6, 11.7, and 11.8 for equations to calculate the amount or analyte or surrogate introduced into the instrument. Calculations are subject to change based on the data reduction software used. Extract concentrations that exceed the upper calibration range must be diluted and reanalyzed or qualified as estimated. Additional internal standard must be added to the diluted extract to maintain the same concentration as the calibration standards.

11.6 When the average response factor from the initial calibration is used, calculate the on-column amount:

$$X_s = \left(\frac{A_s}{RF} \right) x \left(\frac{C_{is}}{A_{is}} \right)$$

11.7 When a linear curve fit is used, calculate the on-column amount:

$$X_s = \left[b + \frac{\left(\frac{A_s}{A_{is}} \right)}{m1} \right] * C_{is}$$

11.8 When a non-linear or quadratic curve fit is used, calculate the on-column amount:

$$X_s = \left[b + m1\left(\frac{A_s}{A_{is}} \right) + m2\left(\frac{A_s}{A_{is}} \right)^2 \right] * C_{is}$$

Where:
RF = Average Response Factor
X_s = On-column concentration of the analyte or surrogate
A_s = Response of the analyte or surrogate in the sample
A_{is} = Response of the internal standard in the sample
C_{is} = Concentration of the internal standard in the sample
m1 = Slope of the line
b = The y-intercept
m2 = coefficient of the determination

11.9 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_t)(D)}{(V_i)(V_s)} \qquad \text{Solid Sample (ug/Kg)} = \frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$$

Where:
X_s = On-column concentration of the analyte in the sample aliquot injected
V_t = Total volume of concentrated extract
D = Dilution factor
V_i = Volume of the extract injected in uL
V_s = Volume of aqueous sample extracted in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{\text{(100- \%Moisture)}} \times 100$$

12. Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits. Refer to Sections 12.1.1 and 12.1.2 for additional information.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) If only one surrogate fails and it is >10% recovery, re-extraction is not required but data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
For BP only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed undiluted or more concentrated. The laboratory may only dilute a sample prior to reanalysis if matrix interference is present. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

12.2 The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS.

12.3 **Allowable Marginal Exceedances**: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: For BP, the LCS shall be allowed to be outside the control limits but ≥10% for hexachlorocyclopentadiene, N-nitrosodimethylamine, pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, and 4-chloroaniline without corrective action. The LCS shall be allowed to be outside the control limits but >10% for up to four additional compounds, with the exception of any PAH, without corrective action.

12.4 LCS equation

$$R = (C/S) * 100$$

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

12.5 MS/MSD equation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.6 RPD calculations:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13. Method Performance

13.1 **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually for each matrix per instrument.

13.2 **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14. Method Modifications

14.1 Standards are purchased as certified stock solutions and not prepared from neat materials.

14.2 Microwave Method 3546 is used for the preparation of solid samples for analysis by 8270C.

14.3 Phenol-d5 is used as a surrogate instead of Phenol-d6.

14.4 Extract final volumes, volume of internal standards added to extracts, and volume of extract injected into the instrument may vary from those identified in Method 8270C.

15. Pollution Prevention and Waste Management

15.1 Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP.

15.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16. References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B, 8000C and 8270C

16.2 Pace Analytical Quality Manual; latest revision.

16.3 TNI Standard; Quality Systems section; latest revision.

17. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1 Table 1: Target Compounds and Reporting Limits

17.2 Table 2: Characteristic Ions of Target Compounds

18. Revisions

Document Number	Reason for Change	Date
S-IN-O-068-rev.10	1. Section 10.13: added language for specific clients that require +/- 15% difference for the ICV. 2. General: revised SOP references where applicable. 3. Section 10.18: added sentence requiring all PAH and surrogate compounds to have a minimum RRF of 0.50 for BP samples. 4. Reworded section 11.10 for clarity.	26Feb2008

S-IN-O-068-rev.11	1. Cover page revised to reflect periodic review format. 2. Table of Contents fixed to include Sections 10 and 11. 3. Section 2: removed reference to Method 3550-Ultrasonic Extraction. 4. Section 3: added language that limits, volumes, etc… are subject to change. 5. Table 7.1: removed TCLP and revised storage conditions. 6. Section 8: revised equipment and supplies where necessary and added "or equivalent" where applicable. 7. Section 9: revised and added "or equivalent" where applicable. 8. Section 10: condensed tuning information and added reference to QAM for ICAL details. Added more detailed equations. 9. Section 11: removed all references to evaluation of the tune mixture for tailing and degradation. Removed instrument conditions tables. Added the calculation for moisture correction. Removed information regarding TICs. 10. Table 12.1: added 1/10 criteria for method blank and reference to Sections 12.1.1 and 12.1.2 for marginal exceedances. 11. Sections 12.1.1 and 12.1.2: added to clarify LCS criteria per NELAC and BP. 12. Section 16: removed references to Tables 3 and 4. 13. Table 2: added internal standard and surrogates to table.	20April2011
S-IN-O-068-rev.12	1. Table of Contents: New Section 14, Method Modifications added. 2. Section 3.1: added a reference to MDLs. 3. Table 7.1: clarified bottle types for both versions of the water extraction. 4. Table 9.3: added detail for the 8270 LVE standards 5. Section 9.2.3: added detail for the 8270 LVE standards. 6. Sections 10.12 and 10.13: revised ICV language to reflect actual practice. 7. Section 11.3: added detail for internal standard addition and analysis of 8270 LVE extracts. 8. Section 11.5: added language for diluting over range samples 9. Sections 11.6-11.8: replaced 8000C equations with Target equations. 10. Table 12.1: revised corrective action for Method Blank. 11. New Section 14 added for Method Modifications 12. Section 15.1: revised SOP reference. 13. Section 16.3: changed NELAC to TNI 14. Attachments Table 1: 1-Methylnaphthalene added. 15. Attachments Table 2: 1-Methylnaphthalene added, Phenol-d6 changed to Phenol-d5.	07May2013

Table 1: Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L)	RL soil (ug/kg)
Phenol	10	330
Bis (2-chloroethyl) ether	10	330
2-Chlorophenol	10	330
Benzyl Alcohol	20	660
2-Methylphenol (o-Cresol)	10	330
Bis (2-chloroisopropyl)ether	5	330
3&4-Methylphenol (m&p-Cresol)	20	660
N-Nitroso-di-n-propylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Bis(2-chloroethoxy)methane	10	330
2,4-Dichlorophenol	10	330
Naphthalene	5	330
4-Chloroaniline	20	660
Hexachlorobutadiene	5	330
4-Chloro-3-methylphenol	20	660
1-Methylnaphthalene	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	10	330
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1600
Dimethyl phthalate	10	330
Acenaphthene	10	330
Acenaphthylene	10	330
2,4-Dinitrophenol	50	1600
4-Nitrophenol	50	1600
Dibenzofuran	10	330
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
3-Nitroaniline	50	1600
Diethyl phthalate	10	330
4-Chlorophenyl phenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	50	1600
4,6-Dinitro-2-methylphenol	50	1600
N-Nitrosodiphenylamine	10	330
4-Bromophenyl phenyl ether	10	330

Analyte	RL water (ug/L)	RL soil (ug/kg)
Hexachlorobenzene	10	330
Pentachlorophenol	50	1600
Phenanthrene	10	330
Anthracene	10	330
Di-n-butyl phthalate	10	330
Fluoranthene	10	330
Pyrene	10	330
Butyl benzyl phthalate	10	330
3,3'-Dichlorobenzidine	20	660
Benzo(a)anthracene	10	330
Chysene	10	330
Bis(2-ethylhexyl)phthalate	5	330
Di-n-octyl phthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-cd)pyrene	10	330
Dibenz(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

[1] Target Compounds and Reporting Limits are subject to change.

Table 2: Characteristic Ions of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
Bis (2-chloroethyl) ether	93	63, 95
2-Chlorophenol	128	64, 130
Benzyl Alcohol	108	77, 79
2-Methylphenol (o-Cresol)	107	77, 108
Bis (2-chloroisopropyl)ether	45	77, 121
3&4-Methylphenol (m&p-Cresol)	107	77, 108
N-Nitroso-di-n-propylamine	70	42, 101
Hexachloroethane	117	199, 201
Nitrobenzene	77	65, 123
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	122	107, 121
Bis(2-chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	98, 164
Naphthalene	128	127, 129
4-Chloroaniline	127	65, 129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	142, 144
1-Methylnaphthalene	142	141
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	165, 194
Acenaphthene	154	152, 153
Acenaphthylene	152	151, 153
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	139	65, 109
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
3-Nitroaniline	138	92, 108
Diethyl phthalate	149	150, 177
4-Chlorophenyl phenyl ether	204	141, 206
Fluorene	166	165, 167
4-Nitroaniline	138	65, 108
4,6-Dinitro-2-methylphenol	198	51, 105
N-Nitrosodiphenylamine	169	167, 168
4-Bromophenyl phenyl ether	248	141, 250

Analyte	Primary Ion	Secondary Ion(s)
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	176, 179
Anthracene	178	176, 179
Di-n-butyl phthalate	149	104, 150
Fluoranthene	202	101, 203
Pyrene	202	200, 203
Butyl benzyl phthalate	149	91, 206
3,3'-Dichlorobenzidine	252	126, 254
Benzo(a)anthracene	228	226, 229
Chysene	228	226, 229
Bis(2-ethylhexyl)phthalate	149	167, 279
Di-n-octyl phthalate	149	43, 167
Benzo(b)fluoranthene	252	125, 253
Benzo(k)fluoranthene	252	125, 253
Benzo(a)pyrene	252	125, 253
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Internal Standards	**Primary Ion**	**Secondary Ion(s)**
1,4-Dichlorobenzene-d4	152	150, 115
Naphthalene-d8	136	68
Acenaphthene-d10	164	160, 162
Phenanthrene-d10	188	80, 94
Chrysene-d12	240	120, 236
Perylene-d12	264	260, 265
Surrogates	**Primary Ion**	**Secondary Ion(s)**
Phenol-d5	99	42, 71
2-Fluorophenol	112	64, 77
Nitrobenzene-d5	82	54, 128
2-Fluorobiphenyl	172	171
2,4,6-Tribromophenol	330	141, 332
Terphenyl-d14	244	122, 212

[2] Target Compounds are subject to change.

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STANDARD OPERATING PROCEDURE

MICROWAVE EXTRACTION
REFERENCE METHOD: EPA SW-846 METHOD 3546

SOP NUMBER:	S-IN-O-130-rev.04
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-O-130-rev.03

APPROVAL

Karl Anderson
General Manager — May 24, 2013 Date

Bud Schrage
Quality Manager — May 16, 2013 Date

Steven R Long
Department Manager — May 16, 2013 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for extracting non-volatile and semi-volatile organic compounds from solid samples, such as soil and sediment, using a microwave extraction system while meeting the requirements specified in EPA method 3546.

2. Summary of Method

2.1. An aliquot of sample is weighed and loaded into an extraction vessel and the appropriate solvent mixture is added and the vessel is sealed. The vessel is heated to the extraction temperature and extracted for a pre-determined length of time. The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined. The extract is concentrated and exchanged as necessary.

3. Scope and Application

3.1. This procedure is for extracting water insoluble or slightly water soluble organic compounds from soils, sediments or other solid waste materials.

3.2. This procedure is applicable to the extraction of semi-volatile compounds including BNAs, PAHs, PCBs, and Diesel Range Organics (DRO).

3.3. This method uses a solvent mixture of 3:1 methylene chloride to acetone. Other solvent systems may be used provided adequate performance can be demonstrated for the target analytes.

3.4. Applicable compounds, volumes/weights, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.5. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of microwave extraction equipment and reagents. Each analyst work cell must demonstrate their capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Solvents, reagents and glassware can all contribute to compound artifacts or elevated baselines; both conditions that can affect chromatography. Method blanks must be analyzed to determine the presence of contaminants.

4.2. Phthalate esters are common contaminant products in many products in the lab. All plastic products should be avoided when performing this method.

4.3. Extracts that exhibit interferences can be run through a cleanup procedure-see EPA method 3600. Before using a cleanup method, the analyst must run a series of calibration standards through the procedure to ensure that the elution order of compounds remains the same and that no new interferent has been introduced by the cleanup method.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Gloves, lab coats and safety glasses must be used to reduce exposure. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions must be prepared in a hood.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". Gloves, lab coats and safety glasses must be used to reduce exposure. It is recommended that sample containers be opened in a hood whenever possible.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	Glass container with Teflon-lined lid (4oz jar).	None	0°C to 6°C	Samples must be extracted within 14 days of the collection date and must be analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. Equipment and Supplies

8.1. Equipment

Equipment	Vendor	Model / Version	Description / Comments
Microwave Extractor	CEM	MARS Express	or equivalent microwave extractor
Mechanical Capper	CEM		or equivalent
Analytical Balance	AND or equivalent		Capable of measuring to 0.1g
Concentrator apparatus	Zymark	Turbo Vap II	or equivalent concentrator

8.2. Supplies

Item	Vendor	Description
Sample vessels	CEM or equivalent	Teflon, for use in the microwave extraction system
Concentrator tubes	Zymark or equivalent	Graduated for concentration of sample to 1 mL
Autosampler vials	Fisher or equivalent	~2mL, clear glass with aluminum crimp-top seals
Micro-syringes	Hamilton or equivalent	Various sizes
Glass funnels	Fisher or equivalent	
Glass wool	Fisher or equivalent	
Filter paper	Whatman Grade 2	Or equivalent filter paper
Spatulas	Fisher or equivalent	
Pipettes	Fisher or equivalent	Volumetric, Class A, various sizes

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	ASTM Type II water	
Sodium Sulfate	Anhydrous, granular	Fisher/ catalog # S415-200LB or equivalent
Methylene Chloride	High Purity Extraction solvent	MG Scientific #9266-8P or equivalent
Acetone	High Purity Extraction solvent	JT Baker 9254-03 or equivalent
Hexane	High Purity Exchange solvent	JT Baker 9262-03 or equivalent
Solvent Mixture	3:1 Methylene Chloride:Acetone	Prepared using High Purity Extraction solvents
Ottawa sand	Mesh size 20-30	Fisher/ catalog # S23-50 or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, initial calibration verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogate compounds must be added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains method-specific spike compounds.	Same solution can be used for LCS and MS/MSD

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock BNA spike standard	1. Restek; catalog # 31004, B/N spike; 1000ug/mL, or equivalent 2. Restek; catalog # 31014, Acid spike; 2000ug/mL, or equivalent 3. Restek; catalog # 561763, Custom PAH spike; 5000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions.
Working BNA spike standards	Refer to Section 9.2.3.1	Good for 6 months from preparation date	Same as stock standard.
Stock/Working BNA surrogate standard	O2si; catalog # 11004-83-1L; 100mg/L, or equivalent. Use 1mL for each BNA.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions.
Stock/Working Full-list BNA spike standard	NSI Solutions; catalog #WL-408-10; TCL BNA Spike; 100ug/mL or equivalent. Use 1mL for each LCS, MS and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions.

Standard Type	Description	Expiration	Storage
Stock PCB spike standard	Restek; catalog # 32029, Aroclor 1016/1260; 1000ug/mL in hexane, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PCB spike standard	Refer to Section 9.2.3.2	Good for 6 months from preparation date	Same as stock standard.
Stock PCB surrogate standard	1. Restek; catalog # 32028, 2,4,5,6-tetrachloro-m-xylene (TCMX); 200ug/mL in acetone, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PCB surrogate standard	Refer to Section 9.2.3.3	Good for 6 months from preparation date	Same as stock standard.
Stock DRO spike standard	Restek; catalog # 31258, Diesel #2 standard; 50,000ug/mL in methylene chloride, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working DRO spike standard	Refer to Section 9.2.3.4	Good for 6 months from preparation date	Same as stock standard.
Stock DRO surrogate standard	Restek; catalog # 31487, Pentacosane; 10,000ug/mL in methylene chloride, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working DRO surrogate standard	Refer to Section 9.2.3.5	Good for 6 months from preparation date	Same as stock standard.
Stock PAH-SIM spike standard	Restek; catalog # 31622, Cal. Mix 5; 2000ug/mL of each compound in methylene chloride, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PAH-SIM spike standard	Refer to Section 9.2.3.6	Good for 6 months from preparation date	Same as stock standard.
Stock PAH-SIM surrogate standard	Restek; catalog # 31062, B/N surrogate; 5000ug/mL of each compound in methylene chloride, or equivlent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PAH-SIM surrogate standard	Refer to Section 9.2.3.7	Good for 6 months from preparation date	Same as stock standard.
Stock/Working Scan/SIM Combo spike standard	O2si, catalog #114072-06, 10-100ug/mL or equivalent. Use 1mL for each LCS, MS and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Stock/Working Scan/SIM Combo surrogate spike standard	O2si, catalog #114071-06; 10-100ug/mL or equivalent. Use 1mL for each sample, Method Blank, LCS, MS and MSD.	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions

9.2.3. Standard Preparation Procedures

9.2.3.1. Working BNA Spike Standard Preparation

Dilute 2.5mL of the stock Acid spike standard (2000ug/mL), 5mL of the stock B/N spike standard (1000ug/mL) and 1mL of the stock Custom PAH spike (5000ug/mL) to 50mL with acetone for a final concentration of 100ug/mL. Add 1mL of this working standard to each BNA LCS, MS and MSD.

9.2.3.2. Working PCB Spike Standard Preparation

Dilute 2mL of the stock PCB spike standard, Aroclors 1016/1260, (1000ug/mL each) to 200mL with acetone for a final concentration of 10ug/mL. Add 250uL of this working spike to each PCB LCS, MS and MSD.

9.2.3.3. Working PCB Surrogate Standard Preparation

Dilute 1mL of the stock PCB surrogate standard, TCMX (200ug/mL) to 200mL with acetone for a final concentration of 1ug/mL. Add 1mL of this working surrogate to each PCB sample, Method Blank, LCS, MS and MSD.

9.2.3.4. Working DRO Spike Standard Preparation

Dilute 10mL of the stock DRO spike standard (50,000ug/mL) to 200mL with acetone for a final concentration of 2500ug/mL. Add 1mL of this working standard to each DRO/ERO/Ohio mod LCS, MS and MSD.

9.2.3.5. Working DRO Surrogate Standard Preparation

Dilute 7.5mL of the stock DRO surrogate standard (10,000ug/mL) to 500mL with acetone for a final concentration of 150ug/mL. Add 1mL of this working surrogate to each DRO/ERO/Ohio mod sample, Method Blank, LCS, MS and MSD.

9.2.3.6. Working PAH-SIM Spike Standard Preparation

Dilute 5mL of the stock PAH-SIM spike standard (2000ug/mL) to 200mL with acetone for a final concentration of 50ug/mL. Add 200uL of this working spike to each PAH-SIM LCS, MS and MSD.

9.2.3.7. Working PAH-SIM Surrogate Standard Preparation

Dilute 5.0mL of the stock PAH-SIM surrogate standard (5000ug/mL) to 500mL with acetone for a final concentration of 50ug/mL. Add 200uL of this working surrogate to each PAH-SIM sample, Method Blank, LCS, MS and MSD.

10. Calibration

10.1. Not applicable to this SOP.

11. Procedures

11.1. Configure the microwave extraction unit per the manufacturer's instructions.

11.2. Make sure that all glassware and Teflon tubes used for this procedure have been properly cleaned. All washed glassware and Teflon tubes must be rinsed prior to use with acetone to remove residual water and with methylene chloride to remove any residual contaminants.

11.3. Decant and discard any water layer on the sample and remove foreign objects such as sticks, leaves, rocks, etc. Sample must be homogenized as much as possible prior to weighing.

11.4. Weigh a 30g portion of sample into an extraction vessel.

11.5. Prepare a method blank and an LCS using 30g portions of Ottawa sand or other clean matrix. The method blank will be used to detect potential contamination. The LCS will be used to determine the efficiency of the extraction method in recovering target compounds.

11.6. Choose one sample in the batch to use as a matrix spike (MS) and matrix spike duplicate (MSD). Weigh two additional 30g portions of the sample for the MS and MSD.

11.7. Add the appropriate surrogate solution to each method blank, sample, LCS, MS and MSD. Add the appropriate spiking solution to the LCS, MS and MSD.

11.8. Add 25mL of the appropriate solvent to each sample vessel and seal the vessels tightly using a mechanical capper.

11.9. Place all extraction vessels into the extraction unit and proceed with microwave extraction per manufacturer's instructions. Once established, the same extraction conditions must be used for all method blanks, client samples, and QC samples in the same extraction batch.

11.10. Allow the extracts to cool to room temperature once the extraction process is complete. After cooling, carefully open the extraction vessels using the mechanical capper and filter each extract through a funnel fitted with filter paper or a glass wool plug and containing sodium sulfate. Collect the extract in a clean, labeled concentrator tube. Rinse extraction vessel with methylene chloride and pour into funnel. Once the extract and rinse is filtered, rinse the sodium sulfate with methylene chloride and collect.

11.11. Concentration procedure: Place each concentrator tube into the Zymark concentrator apparatus. Adjust the settings per manufacturer's instructions. When the volume of the extract reaches a level slightly below the required final volume, remove the concentrator tube from the apparatus and allow extract to cool. Required final volumes are 1mL for BNA, DRO, Scan/SIM Combo and PAH-SIM, and 10mL for PCBs.

11.12. Solvent exchange is required for PCB analysis. When PCB extract has been concentrated to 1mL, add 50mL of hexane to the concentrator tube. Continue to concentrate the extract to slightly below 10mL on the Zymark concentration apparatus. Remove the extract from the water bath and allow extract to cool.

11.13. Prepare a calibrated vial by volumetrically dispensing the required volume of the solvent being used into a vial and securely capping the vial to eliminate evaporation. The calibrated vial must be prepared daily. Quantitatively transfer the extract from the Zymark tube or concentrator tube to a vial. Bring the sample extract in the vial to the required final volume listed in 11.11 by visually comparing the sample extract vial volume to the calibrated vial volume. Securely cap the sample extract vial. Store all extracts in the appropriate storage cooler. For extracts that will not concentrate to the usual final volume, use the procedure described above to bring the extract to the next higher practical volume for which a calibrated vial can be prepared.

11.14. Refer to Sulfuric Acid Cleanup for PCBs SOP and/or Sulfur Cleanup for PCBs Copper Method SOP as required for extract cleanup procedures.

12. Quality Control

12.1. Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Sodium sulfate or Ottawa sand	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, affected data must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to LIMS for control limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to LIMS for control limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to LIMS for control limits.	Samples with surrogate failures must be re-extracted and reanalyzed. QC samples with surrogate failures require the re-extraction and reanalysis of the QC samples and the associated client samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) If only one surrogate for BNA or Combo fails and it is >10% recovery, re-extraction is not required but data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

12.2. LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

12.3. MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4. RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13. Method Performance

13.1. Method Detection Limit (MDL) Study: An MDL study must be conducted every 12 months for each matrix per instrument.

13.2. Demonstration of Capability (DOC): Analysts performing this method must document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

13.3. Microwave Power Test: The microwave should be power tested per manufacturer's instructions monthly, at a minimum.

14. Method Modifications

14.1. Samples not air dried and ground nor are they mixed with sodium sulfate prior to extraction.

14.2. Balance weighs to 0.1g instead of 0.01g because dry weight is not determined by this procedure.

14.3. Samples are not particle sized or sieved prior to extraction.

15. Pollution Prevention and Waste Management

15.1. Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

15.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16. References

16.1. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods"; EPA SW-846, latest revision. Method 3546 "Microwave Extraction".

16.2. Sulfuric Acid Cleanup for PCBs SOP and Sulfur Cleanup for PCBs Copper Method SOP as applicable.

16.3. Pace Analytical Quality Manual; latest revision.

16.4. TNI Standard; Quality Systems section; latest revision.

17. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1. Not applicable to this SOP.

18. Revisions

Document Number	Reason for Change	Date
S-IN-O-130-rev.1	1. General: revised SOP references where applicable. 2. Table 12.1: added prep batch time change (for BP only) to 8 hours. Corrected method blank component to sodium sulfate. 3. Section 9.2.1: added information about standard verification (80-120% limits) per BP requirements (plus allowance for poor performers). 4. Section 11.7: added solvent mixture used.	25Feb2008
S-IN-O-130-rev.2	1. Table 7.1: changed 8oz. jar to 4oz. jar. 2. Section 8.1: added mechanical capper to the equipment list. Added "or equivalent" 3. Section 8.2: removed glass beakers from the supplies list. Added "or equivalent". 4. Section 9.1: added "or equivalent". 5. Table 9.2: clarified spiking standard description. 6. Table 9.3: added "or equivalent" and removed all pesticide and EPH references. 7. Section 9.2.3: removed all pesticide and EPH references. Revised working SIM surrogate standard preparation. 8. Section 11.1: revised microwave settings. Added required final volumes. Clarified procedure for bringing extracts to required final volume. 9. Table 12.1: clarified MS/MSD frequency for BP samples	15May2009
S-IN-O-130-rev.03	1. Table of Contents fixed to include sections 10 and 11. 2. Section 3: language added that volumes/weights, vendors, etc… are subject to change. 3. Table 7.1: updated/clarified storage and hold time requirements 4. Section 8.2: added filter paper 5. Section 9: revised with updated information and removed TCLP references. 6. Section 11: removed microwave settings and referred to manufacturer's instructions. Added reference to filter paper and Scan/SIM Combos. Updated final extract determination technique to the comparison vial method. 7. Table 12.1: updated and clarified exceptions. Referred to LIMS for control limits. 8. Section 13: added reference to microwave power testing. 9. Section 15: changed NELAC to TNI.	03May2011

S-IN-O-130-rev.04	1. Table of Contents: new Section 14, Method Modifications added. 2. Table 8.1: updated balance details 3. Table 8.2: added volumetric pipettes 4. Table 9.1: updated solvent manufacturers 5. Table 9.3: updated for current standards, vendors and concentrations 6. Section 9.2.3: updated for current standards and volumes 7. Section 11.2: added Teflon tubes require washing and rinsing 8. Section 11.8: changed specific solvent to "appropriate solvent" 9. Section 11.11: updated final volumes for methods where needed 10. Section 11.13: added instructions for determination of non-standard final volumes 11. Section 11.14: added reference to cleanup SOPs. 12. Table 12.1: updated corrective action for Method Blank. 13. New Section 14, Method Modifications added. 14. Section 15.1: revised SOP reference. 15. Section 16: added reference to cleanup SOPs.	16May2013

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STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS SELECTIVE ION MONITORING (SIM)

REFERENCE METHOD: EPA SW-846 METHOD 8270C

SOP NUMBER:	S-IN-O-133-rev.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-O-133-rev.02

APPROVAL

General Manager	March 23, 2012 Date
Quality Manager	March 21, 2012 Date
Department Manager	March 21, 2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose

1.1 The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of semi-volatile organic compounds in sample extracts while meeting the requirements specified in EPA method 8270C, through selective ion monitoring (SIM).

2. Summary of Method

2.1. Semi-volatile compounds are introduced into a gas chromatograph by injection of a sample extract onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes that are then detected with a mass spectrometer. Identification of the analytes is made by comparing their mass spectra with spectra of known standards. Quantitation is accomplished by comparing the response of a major ion relative to the internal standard response using a multi-point calibration curve. Selective ion monitoring (SIM) is the mode of scanning for this SOP.

2.2. Method 8270C SIM provides chromatographic conditions for the detection of semi-volatile compounds in organic extracts of groundwater, surface water, soil and sediment. Aqueous samples are extracted by using SW-846 method 3510 Separatory Funnel Extraction or other applicable method. Solid samples are extracted using SW-846 method 3546 Microwave Extraction or other applicable method.

3. Scope and Application

3.1. The full list of compounds and reporting limits analyzed by this laboratory for method 8270C SIM, is found in Table 1. Other analytes may be analyzed by this method but must have quality control documentation to support the performance of this method for those analytes.

3.2. Reporting limits, control limits, volumes used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS equipment and the interpretation of GC/MS SIM data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Glassware for the preparatory steps for this method should be thoroughly cleaned and rinsed. Soap products can leave phthalates on the glassware that may interfere chromatographically.

5. Safety

5.1. Standards and Reagents

The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. The use of gloves, lab coats and safety glasses is required. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock semi-volatile standards are toxic and should be handled with extreme care.

5.2. Samples

Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

6. Definitions

Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber glass container with Teflon-lined lid, preferably 1L or 100mL widemouth.	None required	0°C to 6°C	Sample must be extracted within 7 days of collection date and extract must be analyzed within 40 days of extraction date.
Solid	200 grams in 4oz glass jar	None required	0°C to 6°C	Must be extracted within 14 days of collection date and analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples. Sample extracts must be stored in the refrigerator or freezer until analysis is complete.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatographs	Hewlett Packard	Lab uses models 6890 and 7890	Or equivalent system
Data Systems	Hewlett Packard		Or equivalent system
Autosamplers	Hewlett Packard		Or equivalent system
Mass Spectrometers	Hewlett Packard	Lab uses models 5973 and 5975	Must be capable of operating in **SIM** mode. Or equivalent system.

8.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Columns	Restek	Rtx-5MS	15m x 0.25mm, or equivalent column

8.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials (clear vials)	Supelco or equivalent
Autosampler vials	1.8mL clear vials	Or equivalent

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	Pesticide grade or equivalent
Methanol	Pesticide grade or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions

Standard	Description	Comments
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains all target analytes and is used to prepare MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

9.2.2. Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
PAH-SIM Standards			
Stock PAH-SIM calibration standard	Restek; catalog # 31622; 2000ug/mL or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Intermediate PAH-SIM calibration standards 1 and 2	Refer to Sections 9.2.3.1 and 9.2.3.2.	Solution good for 6 months from preparation	Same as stock standard.
Working PAH-SIM calibration standard	Refer to Section 9.2.3.3.	Solution good for 6 months from preparation	Same as stock standard
Stock PAH-SIM ICV standard w/surrogates	NSI Solutions; catalog #WL-509, 200ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working PAH-SIM ICV standard w/surrogates	Refer to Section 9.2.3.4.	Solution good for 6 months from preparation	Same as stock standard
Stock PAH-SIM Surrogate standard	Restek; catalog #31024, 1000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Stock PAH-SIM Internal standard	Restek; catalog #31006, 4000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working PAH-SIM Internal standard	Refer to Section 9.2.3.5.	Solution good for 6 months from preparation	Same as stock standard

PAH-SIM Low Volume Extraction (LVE) Standards			
Intermediate PAH-SIM LVE calibration standards 1 and 2	Refer to Section 9.2.3.6 and 9.2.3.7	Solution good for 6 months from preparation	Same as stock standard.
Working PAH-SIM LVE calibration standards	Refer to Section 9.2.3.8	Solution good for 6 months from preparation	Same as stock standard
Working PAH-SIM LVE ICV standard w/surrogates	Refer to Section 9.2.3.9	Solution good for 6 months from preparation	Same as stock standard
Working PAH-SIM LVE Internal Standard	Refer to Section 9.2.3.10	Solution good for 6 months from preparation	Same as stock standard

9.2.3. Standard Preparation Procedures

9.2.3.1 Intermediate PAH-SIM Calibration Standard 1

Dilute 100uL of the Stock PAH-SIM Calibration Standard (2000ug/mL) plus 200uL of the Stock PAH-SIM Surrogate Standard (1000ug/mL) to 1mL with methylene chloride for a final concentration of 200ug/mL.

9.2.3.2 Intermediate PAH-SIM Calibration Standard 2

Dilute 25uL of the Intermediate PAH-SIM Calibration Standard 1 to 1mL with methylene chloride for a final concentration of 5ug/mL.

9.2.3.3 Working PAH-SIM Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate Standard amount (1 or 2)	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Working Internal Standard amount added to Cal. Std.	Internal Standard Concentration
Calibration Std 1	10uL (Int 2)	1mL	0.05ug/mL	10uL	5ug/mL
Calibration Std 2	20uL (Int 2)	1mL	0.1ug/mL	10uL	5ug/mL
Calibration Std 3	100uL (Int 2)	1mL	0.5ug/mL	10uL	5ug/mL
Calibration Std 4	200uL (Int 2)	1mL	1ug/mL	10uL	5ug/mL
Calibration Std 5	25uL (Int 1)	1mL	5ug/mL	10uL	5ug/mL
Calibration Std 6	50uL (Int 1)	1mL	10ug/mL	10uL	5ug/mL
Calibration Std 7	100uL (Int 1)	1mL	20ug/mL	10uL	5ug/mL
Calibration Std 8	250uL (Int 1)	1mL	50ug/mL	10uL	5ug/mL

9.2.3.4 Working PAH-SIM ICV Standard with Surrogates

Dilute 50uL of the Stock ICV standard (200ug/mL) to 1mL with methylene chloride for a final concentration of 10ug/mL.

9.2.3.5 Working PAH-SIM Internal Standard

Dilute 375uL of the Stock Internal Standard (4000ug/mL) to3mL with methylene chloride for a final concentration of 500ug/mL.

9.2.3.6 Intermediate PAH-SIM LVE Calibration Standard 1

Dilute 10uL of the Stock PAH-SIM Calibration Standard (2000ug/mL) plus 20uL of the Stock PAH-SIM Surrogate Standard (1000ug/mL) to 1mL with methylene chloride for a final concentration of 20ug/mL.

9.2.3.7 Intermediate PAH-SIM LVE Calibration Standard 2

Dilute 2.5uL of the Intermediate PAH-SIM Calibration Standard 1 to 1mL with methylene chloride for a final concentration of 0.5ug/mL.

9.2.3.8 Working PAH-SIM LVE Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate Standard amount (1 or 2)	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Intermediate Internal Standard amount added to Cal. Std.	Internal Standard Concentration
Calibration Std 1	10uL (Int 2)	1mL	0.005ug/mL	10uL	0.5ug/mL
Calibration Std 2	20uL (Int 2)	1mL	0.01ug/mL	10uL	0.5ug/mL
Calibration Std 3	100uL (Int 2)	1mL	0.05ug/mL	10uL	0.5ug/mL
Calibration Std 4	200uL (Int 2)	1mL	0.1ug/mL	10uL	0.5ug/mL
Calibration Std 5	25uL (Int 1)	1mL	0.5ug/mL	10uL	0.5ug/mL
Calibration Std 6	50uL (Int 1)	1mL	1.0ug/mL	10uL	0.5ug/mL
Calibration Std 7	100uL (Int 1)	1mL	2.0ug/mL	10uL	0.5ug/mL
Calibration Std 8	250uL (Int 1)	1mL	5.0ug/mL	10uL	0.5ug/mL
Calibration Std 9	500uL (Int 1)	1mL	10.0ug/mL	10uL	0.5ug/mL

9.2.3.9 Working PAH-SIM LVE ICV Standard with Surrogates

Dilute 5.0uL of the Stock ICV standard (200ug/mL) to 1mL with methylene chloride for a final concentration of 1.0ug/mL.

9.2.3.10 Intermediate PAH-SIM LVE Internal Standard

Dilute 12.5uL of the Stock Internal Standard (4000ug/mL) to1mL with methylene chloride for a final concentration of 50ug/mL.

10. Calibration

10.1. The instrument is auto-tuned periodically according to manufacturer's instructions to maximize detector sensitivity. DFTPP tuning is not performed for this method. DFTPP tune data is acquired in scan mode. For SIM analysis, the instrument is set to selective ion monitoring (SIM) mode. Verification of hardware tuning is accomplished through the analysis of initial and continuing calibration standards in which the known ions are selected for data acquisition.

10.2. **Initial Calibration:** Initial Calibration standards are introduced into the GC from the lowest to highest concentration by direct injection of 1uL of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. The 12 hour clock during which standards and samples may be analyzed

begins with the injection of the first calibration standard. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

10.3. The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

10.4. The percent relative standard deviation (%RSD) of the response factors is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of the RFs for a compound
RF_{avg} = Mean of RFs for a compound

10.5. All reported PAH compounds in the calibration must be evaluated as CCC compounds. Acceptance criteria is $\leq$30% RSD for each compound to be used for quantitation. If RSD is >30% then linearity is proven by linear regression.

10.6. All reported PAH compounds in the calibration must be evaluated as SPCC compounds. Each compound must meet a minimum average response factor of 0.050

10.7. If the percent relative standard deviation (%RSD) of the RFs for a compound is $\leq$15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

10.8. If the %RSD of the RFs for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.9. The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$r = \frac{\sum_{i=1}^{N} (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} (Y_i - \overline{Y})^2\right)}}$$

10.10. Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be greater than or equal to 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$\text{COD} = \frac{\sum_{i=1}^{N} (Y_i - \overline{Y})^2 - (\frac{n-1}{n-p}) \sum_{i=1}^{N} (Y_i - Y_i')^2}{\ldots\ldots\ldots\ldots \sum_{i=1}^{N} (Y_i - \overline{Y})^2}$$

10.11. Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards should also be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.12. Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent drift of the observed ICV response from the expected ICV response, according to the following equation:

$$\% \text{ Drift} = \frac{\text{Calculated concentration} - \text{Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately after the initial calibration curve. All compounds must have a maximum percent drift of +/- 30%. Allowances for poor performers must be approved by the supervisor and plainly documented as to which compounds are affected. **For BP, the ICV must meet the same criteria as the CCV.**

10.13. ICV Corrective Action: If the ICV fails the criteria, another ICV may be analyzed. If the second ICV fails, a new initial calibration curve must be analyzed. Instrument maintenance and or preparation of new calibration standards should also be considered. Samples associated with a failed ICV must be reanalyzed. **Exception:** If the ICV fails during an overnight run and is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

10.14. Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a Continuing Calibration Verification (CCV) standard. The CCV is prepared using the same standard solution used for the initial calibration. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the CCV.

10.15. If the % Difference (%D) or % Drift for each CCC is ≤20%, then the initial calibration is assumed to be valid. Each SPCC must meet the SPCC criteria in Section 10.6. All reported PAH compounds are considered CCCs and SPCCs for the evaluation of the CCV. **For BP, surrogate compounds must also be ≤20%D/Drift.**

$$\% \text{ Difference} = [RF_v - RF_{avg}] / RF_{avg} * 100$$

10.16. The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

10.17. CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11. Procedures

11.1. All sample extracts must be analyzed at room temperature and the system must be calibrated and free of contamination before samples are analyzed.

11.2. **Gas Chromatography conditions**: Configure the GC/MS per manufacturer's instructions.

11.3. The 1mL extract obtained from the sample preparation laboratory should be fortified with 10uL of the Working PAH-SIM or PAH-SIM LVE Internal Standard solution just prior to analysis, resulting in an internal standard concentration of 5ug/mL for PAH-SIM or 0.5ug/mL for PAH-SIM LVE. Analyze each extract by injecting 1uL onto the column.

11.4. **Qualitative Analysis**

11.4.1 The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

11.4.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. See Table 2 for primary and secondary ions.

11.4.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.5. **Quantitative analysis:** Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. See Sections 11.6, 11.7, and 11.8 for equations to calculate the amount or analyte or surrogate introduced into the instrument. Calculations are subject to change based on the data reduction software used.

11.6. When the average response factor from the initial calibration is used, calculate the on-column amount:

$$X_s = (A_s/RF_{avg}) \times (C_{is}/A_{is})$$

Where: X_s = On-column concentration of the analyte or surrogate
A_s = Peak area or height of the analyte or surrogate in the sample
A_{is} = Peak area or heath of the internal standard in the sample
C_{is} = Concentration of the internal standard in the sample
RF_{avg} = The average response factor from the initial calibration

11.7 When a linear curve fit is used, calculate the on-column amount:

$$X_s = \frac{[A_s/A_{is} - b]}{a} \times C_{is}$$

where: A_s = Peak area or height of the analyte or surrogate in the sample
A_{is} = Peak area or height of the internal standard in the sample
X_s = On-column concentration of the analyte or surrogate
C_{is} = Concentration of the internal standard in the sample
a = Slope of the line
b = The intercept

11.8 When a non-linear or quadratic curve fit is used, calculate the on-column amount:

$$X_s = \frac{-b + \sqrt{b^2 - 4a\left(c - \frac{A_s}{A_{is}}\right)}}{2a} \times C_{is}$$

where: X_s = On-column concentration of the analyte or surrogate
A_s = Peak area or height of the analyte or surrogate in the sample
A_{is} = Peak area or height of the internal standard in the sample
C_{is} = Concentration of the internal standard in the sample
a = Slope of the line
b = The intercept
c = coefficient of the polynomial

11.9 Calculate the final concentration in the sample as follows:

$$\text{Aqueous Sample (ug/L)} = \frac{(X_s)(V_t)(D)}{(V_i)(V_s)} \qquad \text{Solid Sample (ug/Kg)} = \frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$$

Where: X_s = On-column concentration of the analyte in the sample aliquot injected
V_t = Total volume of concentrated extract
D = Dilution factor
V_i = Volume of the extract injected in uL
V_s = Volume of aqueous sample extracted in milliliters
W_s = Weight of solid sample extracted in grams

$$\text{Moisture corrected concentration} = \frac{\text{(Final concentration as received)}}{(100 - \%\text{Moisture})} \times 100$$

12. Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, the sample data may be reported without qualifiers, the method blank must be qualified.
Laboratory Control Sample (LCS)	Target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits. Refer to Sections 12.1.1 and 12.1.2 for additional information.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
For BP only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed undiluted or more concentrated. The laboratory may only dilute a sample prior to reanalysis if matrix interference is present. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

12.1.1 The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS.

12.1.2 **Allowable Marginal Exceedances**: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: For BP, marginal exceedances are not allowed. All PAH compounds must be within control limits.

12.2 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13 Method Performance

13.1 **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually per matrix per instrument.

13.2 **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

14 Pollution Prevention and Waste Management

14.1 Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling or other applicable SOP.

14.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15 References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B, 8000C and 8270C

15.2 Pace Analytical Quality Manual; latest revision.

15.3 TNI Standard; Quality Systems section; latest revision.

16 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

16.1 Table 1: Method Compounds and Reporting Limits

16.2 Table 2: Primary and Secondary Ions

17 Revisions

Document Number	Reason for Change	Date
S-IN-O-133-rev.1	1. Section 10.11: added note about client-specific ICV acceptance limits (BP, Marathon, Speedway). 2. General: revised SOP references where applicable. 3. Section 10.6: added minimum response factors to use as SPCC criteria (per Marathin requirement from CLP SOW). 4. Section 10.15: referenced new SPCC criteria in section 10.6. 5. Removed section 11.10-11.12 (TICs- not applicable to SIM). 6. Removed old sections 3.3 and 3.4 (not applicable).	19Mar2008
S-IN-O-133-rev.02	1. Title page converted to periodic review format 2. Table of Contents fixed to contain Sections 10 and 11 3. Section 2.2: reference to Method 3550 removed. 4. Section 3: added language that limits, volumes, etc… are subject to change. 5. Table 7.1: removed TCLP and revised storage conditions. 6. Section 8: revised equipment and supplies where necessary and added "or equivalent" where applicable. 7. Section 9: revised and added "or equivalent" where applicable. 8. Section 10: added more detailed equations and revised SPCC criteria to match 8270C. Clarified ICAL criteria. 9. Section 11: removed table with instrument conditions and added the calculation for moisture correction. 10. Table 12.1: added 1/10 criteria for method blank and reference to Sections 12.1.1 and 12.1.2. 11. Sections 12.1.1 and 12.1.2 added to clarify LCS criteria. 12. Section 15: revised references to include 8000C.	18Mar2011
S-IN-O-133-rev.03	1. Table 7.1: added sample container for reduced sample volume extraction or PAH-SIM LVE 2. Table 9.3: added standards for PAH-SIM LVE. 3. Section 9.2.3: added preparation instructions for PAH-SIM LVE standards. 4. Section 11.3: added internal standard information for PAH-SIM LVE. 5. Section 14.1: revised SOP reference. 6. Section 15.3: changed NELAC to TNI	20Mar2012

Table 1
Method 8270C SIM Compounds and Reporting Limits[1]

Analyte	RL aqueous (ug/L) 8270C SIM	RL solid (ug/kg) 8270C SIM
Acenaphthene	1	25
Acenaphthylene	1	25
Anthracene	0.1	25
Benzo(a)anthracene	0.1	25
Benzo(a)pyrene	0.1	25
Benzo(b)fluoranthene	0.1	25
Benzo(g,h,i)perylene	0.1	25
Benzo(k)fluoranthene	0.1	25
Chrysene	0.5	25
Dibenz(a,h)anthracene	0.1	25
Fluoranthene	1	25
Fluorene	1	25
Indeno(1,2,3-cd)pyrene	0.1	25
1-Methylnaphthalene	1	25
2-Methylnaphthalene	1	25
Naphthalene	1	25
Phenanthrene	1	25
Pyrene	1	25

[1] Reporting Limits are subject to change.

Table 2
8270C SIM Primary and Secondary Ions

Analytes	Primary Ion	Secondary Ion
Acenaphthene	154	153
Acenaphthylene	152	151
Anthracene	178	176
Benzo(a)anthracene	228	229
Benzo(a)pyrene	252	253
Benzo(b)fluoranthene	252	253
Benzo(g,h,i)perylene	276	138
Benzo(k)fluoranthene	252	253
Chysene	228	226
Dibenz(a,h)anthracene	278	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	200
Internal Standards	**Primary Ion**	**Secondary Ion**
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162
Phenanthrene-d_{10}	188	94
Chrysene-d_{12}	240	120
Perylene-d_{12}	264	260
Surrogates	**Primary Ion**	**Secondary Ion**
2-Fluorobiphenyl	172	171
Terphenyl-d_{14}	244	122

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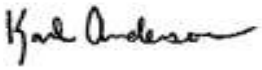
STANDARD OPERATING PROCEDURE

Extraction of Oils and Wipes for PCB Analysis

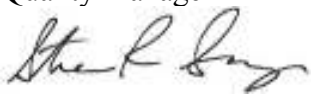
SOP NUMBER:	S-IN-O-161-rev. 02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-O-161-rev. 1

APPROVAL



General Manager — April 20, 2012 — Date

Quality Manager — April 11, 2012 — Date



Department Manager — April 11, 2012 — Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

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1. PURPOSE

1.1 The purpose of this Standard Operating Procedure (SOP) is to describe the preparation of oils and gauze wipes for analysis of PCBs by SW-846 Method 8082.

2. SUMMARY OF METHOD

2.1 For each oil sample, 1g of oil is diluted into 10mL of Hexane. The diluted sample may be subjected to one or more cleanup procedures prior to analysis.

2.2 For each wipe samples, the following are sent to the client: A 4-oz pre-cleaned jar containing a gauze wipe and 50mL hexane. The lab will extract the sample directly in this jar with hexane.

2.3 Spike amounts, sample weights and extract final volumes may be adjusted to achieve project-specific reporting limits.

3. SCOPE AND APPLICATION

3.1 These procedures describe a solvent dilution of oil samples and a solvent extraction of wipe samples prior to cleanup and/or analysis.

3.2 Applicable volumes/weights, standard concentrations, vendors, equipment and supplies are subject to change.

3.3 This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of listed equipment and reagents. Each analyst work cell must demonstrate their capability to generate acceptable results with this method to be considered qualified to report sample results.

4. INTERFERENCES

4.1 Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that can lead to discrete artifacts and/or elevated baselines. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks (Method Blanks). Contact with common plastics or rubber products must be avoided.

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.

5. SAFETY

5.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Gloves, lab coats and safety glasses must be used to reduce exposure. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions must be prepared in a hood.

5.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous “unknowns”. Gloves, lab coats and safety glasses must be used to reduce exposure. It is recommended that sample containers be opened in a hood whenever possible.

6. DEFINITIONS

6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Oils	Glass container with Teflon-lined lid (4oz jar).	None	0°C to 6°C	Samples must be extracted within 14 days of the collection date and must be analyzed within 40 days of extraction date.
Wipes	Glass container with Teflon-lined lid (4oz jar).	None	0°C to 6°C	Samples must be extracted within 14 days of the collection date and must be analyzed within 40 days of extraction date.

Samples must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

8. EQUIPMENT AND SUPPLIES

8.1 Equipment

Equipment	Vendor	Description / Comments
Balance	OHaus or equivalent	Capable of measuring to 0.1g
Concentrator apparatus	Zymark Turbo Vap II	or equivalent concentrator
Shaker table	Eberbach 6010	or equivalent equipment

8.2 Supplies

Item	Description
Sample jars	Pre-cleaned, 200 series or higher, Teflon lined closure or equivalent
Gauze pads	12-ply non-woven, 3”x3”, or equivalent wipes
Concentrator tubes	Graduated for concentration of sample to 1 mL
Autosampler vials	2mL, clear glass with aluminum crimp-top seals
Sample vials	20mL with Teflon lined closure, or equivalent
Micro-syringes	Various sizes, gas tight or equivalent
Glass funnels	
Glass wool	
Filter paper	Or equivalent filter paper
Pasteur pipettes	Disposable

9. REAGENTS AND STANDARDS

9.1 Reagents

Reagent	Concentration/ Description
Sodium Sulfate	Anhydrous, granular
Hexane	Pesticide grade or equivalent
PCB-free Transformer Oil	Restek cat #32425, for use as a simulated matrix, or equivalent substitute

9.2 Analytical Standards

9.2.1 Definitions

Standards are required for initial calibration, initial calibration verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Surrogate standard	Surrogate compounds must be added to each sample and QC sample to monitor extraction efficiency.	
Spiking Standard	This solution contains method-specific spike compounds.	Same solution can be used for LCS and MS/MSD

9.2.2 Storage Conditions

Table 9.3 – Analytical Standard Storage Conditions

Standard Type	Description	Expiration	Storage
Stock PCB spike standard	1. Restek; catalog # 32006, Aroclor 1016; 1000ug/mLin hexane, or equivalent 2. Restek; catalog # 32012, Aroclor 1260; 1000ug/mL in hexane, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PCB spike standard	Refer to Section 9.2.3.1	Good for 6 months from preparation date	Same as stock standard.
Stock PCB surrogate standard	Restek; catalog # 32027, 2,4,5,6-tetrachloro-m-xylene (TCMX); 200ug/mL in acetone, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working PCB surrogate standard	Refer to Section 9.2.3.2	Good for 6 months from preparation date	Same as stock standard.

9.2.3 Standard Preparation Procedures

9.2.3.1 Working PCB Spike Standard Preparation

Dilute 1mL of each PCB stock standard, Aroclors 1016 and 1260, (1000ug/mL each) to 200mL with acetone for a final concentration of 5ug/mL. Add 1mL of this working spike to each PCB LCS, MS and MSD.

9.2.3.2 Working PCB Surrogate Standard Preparation

Dilute 5mL of the PCB stock surrogate standard, TCMX (200ug/mL) to 500mL with acetone for a final concentration of 2ug/mL. Add 1mL of this working surrogate to each PCB sample, Method Blank, LCS, MS and MSD.

10. CALIBRATION

10.1 Not applicable.

11. PROCEDURE

11.1 Oil Dilution

11.1.1 Prepare a calibrated vial by volumetrically dispensing 10mLs of hexane into a 20mL vial and securely capping the vial to eliminate evaporation. The calibrated vial must be prepared daily.

11.1.2 Weigh 1g of the oil sample into a clean 20mL vial. If requested or required, prepare

additional sample vials for a Matrix Spike and Matrix Spike Duplicate.

11.1.3 Prepare method blank and LCS by labeling clean 20mL vials. Weigh 1g of PCB-free Transformer oil into each vial.

11.1.4 Spike 1mL of Working PCB Surrogate Standard (2ug/mL) into each vial. For LCS or Matrix Spike, also spike 1mL of the Working PCB Spike Standard (5µg/mL) into the vial.

11.1.5 Bring each vial to 10mL volume with hexane by visually comparing the vial volume to the calibrated vial volume. Securely cap all vials and agitate to mix the contents. Perform cleanup of extracts as necessary. Store all extracts in the appropriate storage cooler.

11.2 Wipe Extraction

11.2.1 Set up and label Turbovap tubes on rack for all samples plus Method Blank, LCS. Place funnels with filter and Sodium Sulfate on top of them. Rinse Sodium Sulfate with hexane into Turbovap tubes, rinse tubes, and discard rinse solvent.

11.2.2 Prepare a method blank and LCS using a clean gauze wipe placed into a 4 oz jar. Sample wipes will be extracted in the jar they are received in by the laboratory. Sample jars must contain enough hexane to completely cover the wipe, approximately 50 mL.

11.2.3 Spike 1mL of Working PCB Surrogate Standard (2µg/mL) onto method blank, LCS and all sample wipes. For LCS, also spike 1mL of the Working PCB Spike Standard (5µg/mL) onto the wipe.

11.2.4 Add 50 mL Hexane to method blank and LCS containers. Add enough additional Hexane to sample wipe containers to bring the solvent volume to approximately 50mLs.

11.2.5 Cap all containers and secure onto shaker table. Shake on high setting for 3 minutes.

11.2.6 Decant extracts through funnels with Sodium Sulfate into labeled Turbovap tubes.

11.2.7 Add 50 mLs Hexane to all jars and shake again for 3 minutes. Decant through funnels. Repeat this process for a third time for all containers.

11.2.8 Place the extracts contained in the Turbovap tubes into the Zymark concentrator. Concentrate the extracts to just below 10mLs. Remove the extracts from the water bath and allow them to cool.

11.2.9 Prepare a calibrated vial by volumetrically dispensing 10mLs of hexane into a vial and securely capping the vial to eliminate evaporation. The calibrated vial must be prepared daily. Quantitatively transfer the extract from the Zymark tube to a vial. Bring the extract in the vial to 10mLs by visually comparing the extract vial volume to the calibrated vial volume. Securely cap the sample extract vial. Perform cleanup of extracts as necessary. Store all extracts in the appropriate storage cooler.

12. QUALITY CONTROL

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Simulated matrix	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, the sample data may be reported without qualifiers, the method blank must be qualified.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to LIMS for control limits.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported without qualifiers. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD) or Sample Duplicate (DUP)	Applicable target analytes	As requested or required by client.	Lab-generated limits Refer to LIMS for control limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD or DUP must be qualified appropriately.
Surrogate	Applicable surrogate compound	Added to each sample, standard and method blank	Lab-generated limits Refer to LIMS for control limits.	Samples with surrogate failures must be re-extracted and reanalyzed. QC samples with surrogate failures require the re-extraction and reanalysis of the QC samples and the associated client samples. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported without qualifiers. The surrogate must be qualified. 3) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.

12.2 LCS equation:

$$R = (C/S) * 100$$

Where R = percent recovery
C = spiked LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation:

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD equation:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13. METHOD PERFORMANCE

13.1 Demonstration of Capability (DOC): Analyst work cells performing this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC).

13.2 A Method Detection Limit (MDL) Study must be performed annually for Aroclors 1016/1260 at a minimum, per matrix.

13.3 Modifications to this method may be necessary to accommodate non-routine samples, wipe dimensions or sampling procedures.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in S-ALL-S-002, Waste Handling or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

14.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. REFERENCES

15.1 Verification of PCB Spill Cleanup by Sampling and Analysis, EPA-560/5-85-026, August, 1985.

15.2 USEPA, SW-846, Method 3580A, Revision 1, July 1992.

15.3 TNI Standard, Quality Systems section, latest revision.

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ETC.

16.1 Not Applicable

17. REVISIONS

Document Number	Reason for Change	Date
S-IN-O-161-rev.0	First issue of SOP	28Aug2009
S-IN-O-161-rev.1	1. Cover page revised for periodic review instead of annual review. 2. Table of Contents format revised 3. Sections 11.1.8 and 11.1.9: revised to new final volume procedure. 4. Section 13.4: revised to not require MDL if not reporting below RL 5. Section 15: removed references to cleanup SOPs	16Mar2010
S-IN-O-161-rev.02	1. Cover page: revised title to include Oil and updated periodic review section. 2. Section 1.1: added reference to oil samples. 3. Section 3: added summary of oil extraction and flexibility in volumes and spike amounts. 4. Section 5.2: removed SOP reference. 5. Section 7: updated information and changed to tabular format 6. Section 8: updated information and changed to tabular format 7. Section 9: updated information and changed to tabular format 8. Section 11: added an entire section (11.1) regarding extraction of oils. 9. Section 12: updated by inserting standard format and content and adding calculations for LCS, MS/MSDand RPD. 10. Sections 13 and 14: updated to be consistent with standard SOP formatting and content. 11. Section 15: added reference to 3580A and TNI Standard, removed reference to 8082A and Pace SOP.	11Apr2012

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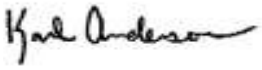
STANDARD OPERATING PROCEDURE

THE DETERMINATION OF SEMI-VOLATILE COMPOUNDS BY COMBINATION GC/MS SCAN AND SIM

REFERENCE METHOD: EPA SW-846 METHOD 8270C

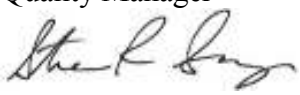
SOP NUMBER:	S-IN-O-163-rev.01
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-IN-O-163-rev.00

APPROVAL



General Manager — May 24, 2013 Date

Quality Manager — May 16, 2013 Date



Department Manager — May 16, 2013 Date

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1. Purpose

1.1. The purpose of this SOP is to provide a laboratory specific procedure for determining the concentration of semi-volatile organic compounds in sample extracts while meeting the requirements specified in EPA method 8270C.

2. Summary of Method

2.1. Samples undergo a solvent extraction and the resulting extract is divided into two equal portions. Semi-volatile compounds are introduced into a gas chromatograph by injection of the sample extract onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes that are then detected with a mass spectrometer. Identification of the analytes is made by comparing their mass spectra with spectra of known standards using both Scan mode and Selective Ion Monitoring (SIM) mode. Quantitation is accomplished by comparing the response of a major ion relative to the internal standard response using a multi-point calibration curve.

2.2. Method 8270C provides chromatographic conditions for the detection of semi-volatile compounds in organic extracts. Aqueous samples are extracted using SW-846 method 3510 Separatory Funnel Extraction or other applicable method.

3. Scope and Application

3.1. The list of compounds and reporting limits analyzed by this laboratory for method 8270C using both Scan and SIM modes is found in Table 1. Other compounds may be reported upon completion of appropriate validation procedures. Refer to the LIMS for method detection limits.

3.2. This method using Scan mode can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, phenols, and nitrophenols. This method using SIM mode can be used to quantitate a subset of semi-volatile compounds such as polynuclear aromatic hydrocarbon (PAH) compounds to achieve lower limits of detection. Other compounds may also be analyzed in SIM mode.

3.3. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent extraction and exhibits poor chromatographic behavior. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosdiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3.4. Reporting limits, control limits, volumes/weights used, standard concentrations, vendors, instrumentation, equipment and supplies are subject to change.

3.5. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of GC/MS equipment and the interpretation of GC/MS data acquired using both Scan and SIM modes. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Interferences

4.1. Glassware for the preparatory steps for this method should be thoroughly cleaned and rinsed. Soap products can leave phthalates on the glassware that in turn show up in the analytical data. Hits for phthalates should be closely scrutinized and continuous hits should warrant checking on the glassware cleaning procedure.

5. Safety

5.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. The use of gloves, lab coats and safety glasses is required. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible. The stock semi-volatile standards are toxic and should be handled with extreme care.

5.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment such as gloves**,** lab coats and safety glasses is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible. All distillations should be conducted under a fume hood.

6. Definitions

6.1. Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions.

7. Sample Collection, Preservation, and Handling

Table 7.1 – Sample Collection, Preservation, Storage and Hold time.

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	Amber Glass container with Teflon-lined lid, preferably 1L or 125mL, widemouth, or equivalent	None required	0°C to 6°C	Must be extracted within 7 days of collection date and analyzed within 40 days of extraction date

Samples and sample extracts must be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

Sample extracts must be stored in the refrigerator or freezer until analysis is complete.

8. Equipment and Supplies

8.1. Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
Gas Chromatographs	Hewlett Packard/Agilent	Lab uses models 6890 and 7890	Or equivalent system
Data Systems	Hewlett Packard/Agilent	Chemstation	Or equivalent system
Autosamplers	Hewlett Packard/Agilent		Or equivalent system
Mass Spectrometers	Hewlett Packard/Agilent	Lab uses models 5973 and 5975	Must be capable of operating in **SIM** mode. Or equivalent system

8.2. Chromatography Supplies

Item	Vendor	Model / ID	Description
Analytical Column - Scan	Fisher	J&W Scientific DB5MS or equivalent	15m x 0.25mm or equivalent column
Analytical Column – SIM	Restek	Rtx-5MS or equivalent	15m x 0.25mm, or equivalent column

8.3. General Supplies

Item	Description	Vendor/ Item # / Description
Gas tight syringes	Various sizes	Hamilton or equivalent
Syringe valves	2-way with Luer ends	Supelco or equivalent
Standard vials	2mL stop/go vials (clear vials)	Supelco or equivalent
Autosampler vials	1.8mL clear vials	Or equivalent

9. Reagents and Standards

9.1. Reagents

Reagent	Concentration/ Description
Methylene Chloride	Pesticide grade or equivalent
Acetone	Pesticide grade or equivalent
Methanol	Pesticide grade or equivalent

9.2. Analytical Standards

9.2.1. Definitions

Standards are required for initial calibration, calibration verification standards, second source verification, and for preparing LCS, MS, and MSD samples.

Table 9.2 Standard Definitions and vendors

Standard	Description	Comments
Tuning Standard	Standard used to tune the mass spectrometer in Scan mode. DFTPP tuning is not performed for SIM analysis.	DFTPP solution
Initial Calibration Standards	Standards prepared at varying levels to determine calibration range of the instrument.	
Initial Calibration Verification Standard	A standard prepared from a source other than that used for the initial calibration. This standard verifies the accuracy of the calibration curve.	ICV
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify the initial calibration.	CCV
Spiking Standard	This solution contains method required spiking compounds, at a minimum, and is used for spiking MS/MSD sets.	Same solution can be used for the LCS and MS/MSD

9.2.2. Storage Conditions

Table 9.3a – Analytical Standards and Storage Conditions for 8270 Scan Analysis

Standard Type	Description	Expiration	Storage
Stock 8270/8270 LVE calibration standards	O2si; catalog #110920-01, 1000-2000ug/mL or equivalent O2si; catalog #110931-01, 2000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Intermediate #1 8270 calibration standards	Refer to Section 9.2.3.1	Solution good for 6 months from preparation	Same as stock standard.
Working 8270/Intermediate #2 8270 LVE calibration standards	Refer to Sections 9.2.3.2.	Same expiration date as Intermediate #1 8270 calibration standard	Same as stock standard.
Working 8270 LVE calibration standards	Refer to Sections 9.2.3.3.	Same expiration date as Intermediate #1 8270 LVE calibration standard	Same as stock standard.
Stock 8270 ICV standard with surrogates	NSI; catalog #WL-509, 200ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working 8270/Intermediate 8270 LVE ICV standard	Refer to Section 9.2.3.4.	Solution good for 6 months from preparation	Same as stock standard.
Working 8270 LVE ICV standard	Refer to Section 9.2.3.5.	Same expiration date as Intermediate 8270 LVE ICV standard	Same as stock standard.
Working 8270/Stock 8270 LVE internal standards	Restek; catalog # 31006; 4000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working 8270 LVE internal standards	Refer to Section 9.2.3.6.	Solution good for 6 months from preparation	Manufacturer's recommended storage conditions
Stock 8270/8270 LVE Surrogate standards	Accustandard; catalog # M-8270-SS; 4000ug/mL, or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Stock DFTPP Tuning Standard	Restek; catalog # 31615; 1000ug/mL or equivalent	Manufacturer's recommended expiration date	Manufacturer's recommended storage conditions
Working DFTPP/Intermediate DFTPP LVE Tuning Standard	Refer to Section 9.2.3.7.	Solution good for 6 months from preparation	Same as stock standard
Working DFTPP LVE Tuning Standard	Refer to Section 9.2.3.8.	Same expiration date as Intermediate DFPP LVE Tuning standard	Same as stock standard

Table 9.3b – Analytical Standards and Storage Conditions for 8270 SIM Analysis

Standard Type	Description	Expiration	Storage
Stock 8270-SIM calibration standard	Restek; catalog # 31622; 2000ug/mL or equivalent.	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Intermediate 8270-SIM calibration standards #1 and #2	Refer to Sections 9.2.3.9 and 9.2.3.10.	Solution good for 6 months from preparation	Same as stock standard.
Working 8270-SIM calibration standard	Refer to Section 9.2.3.11.	Solution good for 6 months from preparation	Same as stock standard
Stock 8270-SIM ICV standard w/surrogates	NSI Solutions; catalog #WL-509, 200ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working 8270-SIM ICV standard w/surrogates	Refer to Section 9.2.3.12.	Solution good for 6 months from preparation	Same as stock standard
Stock 8270-SIM Surrogate standard	Restek; catalog #31024, 1000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Stock 8270-SIM Internal standard	Restek; catalog #31006, 4000ug/mL or equivalent	Manufacturer's recommended expiration date.	Manufacturer's recommended storage conditions
Working 8270-SIM Internal standard	Refer to Section 9.2.3.13.	Solution good for 6 months from preparation	Same as stock standard
Working 8270-SIM LVE calibration standards	Refer to Section 9.2.3.14.	Solution good for 6 months from preparation	Same as stock standard
Working 8270-SIM LVE ICV standard w/surrogates	Refer to Section 9.2.3.15.	Solution good for 6 months from preparation	Same as stock standard
Working 8270-SIM LVE Internal Standard	Refer to Section 9.2.3.16.	Solution good for 6 months from preparation	Same as stock standar

9.2.3 Standard Preparation Procedures

9.2.3.1 Intermediate #1 8270 Calibration Standard

Combine 1mL of each Stock 8270 Calibration Standard (1000-2000ug/mL) plus 500uL of the Stock 8270 Surrogate Standard (4000ug/mL) and dilute to 10mL with methylene chloride for a final concentration of 100-200ug/mL.

9.2.3.2 Working 8270 Calibration Standards/Intermediate #2 8270 LVE Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate #1 Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Stock Internal Standard amount added to Cal. Std.	Internal Standard Concentration
8270 Calibration Std 1	2.5uL	500uL	1ug/mL	5uL	40ug/mL
8270 Calibration Std 2	12.5uL	500uL	5ug/mL	5uL	40ug/mL
8270 Calibration Std 3	25uL	500uL	10ug/mL	5uL	40ug/mL
8270 Calibration Std 4	50uL	500uL	20ug/mL	5uL	40ug/mL
8270 Calibration Std 5	125uL	500uL	50ug/mL	5uL	40ug/mL
8270 Calibration Std 6	250uL	500uL	100ug/mL	5uL	40ug/mL
8270 Calibration Std 7	375uL	500uL	150ug/mL	5uL	40ug/mL
8270 Calibration Std 8	437.5uL	500uL	175ug/mL	5uL	40ug/mL
8270 Calibration Std 9	500uL	500uL	200ug/mL	5uL	40ug/mL

9.2.3.3 Working 8270 LVE Calibration Standards

Prepare using the Working 8270 Calibration Standards as Intermediate Standards.
The following are examples of calibration standards and could vary based on requirements:

Standard	Intermediate #2 Cal. Std. ID from Section 9.2.3.2	Intermediate Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Internal Standard Concentration
8270 LVE Cal. Std 1	8270 Calibration Std 1	50uL	500uL	0.1ug/mL	4ug/mL
8270 LVE Cal. Std 2	8270 Calibration Std 2	50uL	500uL	0.5ug/mL	4ug/mL
8270 LVE Cal. Std 3	8270 Calibration Std 3	50uL	500uL	1.0ug/mL	4ug/mL
8270 LVE Cal. Std 4	8270 Calibration Std 4	50uL	500uL	2.0ug/mL	4ug/mL
8270 LVE Cal. Std 5	8270 Calibration Std 5	50uL	500uL	5.0ug/mL	4ug/mL
8270 LVE Cal. Std 6	8270 Calibration Std 6	50uL	500uL	10ug/mL	4ug/mL
8270 LVE Cal. Std 7	8270 Calibration Std 7	50uL	500uL	15ug/mL	4ug/mL
8270 LVE Cal. Std 8	8270 Calibration Std 8	50uL	500uL	17.5ug/mL	4ug/mL
8270 LVE Cal. Std 9	8270 Calibration Std 9	50uL	500uL	20ug/mL	4ug/mL

9.2.3.4 Working 8270 ICV Standard/Intermediate 8270 LVE ICV Standard

Dilute 250uL of the Stock 8270 ICV Standard (200ug/mL) to 500uL with methylene chloride for a final concentration of 100ug/mL.

9.2.3.5 Working 8270 LVE ICV Standard

Dilute 50ul of the Intermediate 8270 LVE ICV Standard (100ug/mL) to 500uL with methylene chloride for a final concentration of 10ug/mL.

9.2.3.6 Working 8270 LVE Internal Standards

Dilute 500uL of the Stock 8270 LVE internal standards (4000ug/mL) to 5mL with methylene chloride for a final concentration of 400ug/mL.

9.2.3.7 Working DFTPP Tuning Standard/Intermediate DFTPP LVE Tuning Standard

Dilute 25uL of the Stock DTPP Tuning Standard (1000ug/mL) to 500uL with methylene chloride for a final DFTPP concentration of 50ng/uL.

9.2.3.8 Working DFTPP LVE Tuning Standard

Dilute 50uL of the Intermediate DTPP LVE Tuning Standard (50ug/mL) to 500uL with methylene chloride for a final DFTPP concentration of 5ng/uL.

9.2.3.9 Intermediate 8270-SIM Calibration Standard #1

Dilute 100uL of the Stock 8270-SIM Calibration Standard (2000ug/mL) plus 200uL of the Stock 8270-SIM Surrogate Standard (1000ug/mL) to 1mL with methylene chloride for a final concentration of 200ug/mL.

9.2.3.10 Intermediate 8270-SIM Calibration Standard #2

Dilute 25uL of the Intermediate 8270-SIM Calibration Standard #1 (200ug/mL) to 1mL with methylene chloride for a final concentration of 5ug/mL.

9.2.3.11 Working 8270-SIM Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	Int. Std amt (#1 or #2)	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Working Internal Standard amount added to Cal. Std.	Internal Standard Concentration
8270-SIM Cal Std 1	10uL (Int #2)	1mL	0.05ug/mL	10uL	5ug/mL
8270-SIM Cal Std 2	20uL (Int #2)	1mL	0.1ug/mL	10uL	5ug/mL
8270-SIM Cal Std 3	100uL (Int #2)	1mL	0.5ug/mL	10uL	5ug/mL
8270-SIM Cal Std 4	200uL (Int #2)	1mL	1ug/mL	10uL	5ug/mL
8270-SIM Cal Std 5 (CCV)	25uL (Int #1)	1mL	5ug/mL	10uL	5ug/mL
8270-SIM Cal Std 6	50uL (Int #1)	1mL	10ug/mL	10uL	5ug/mL
8270-SIM Cal Std 7	100uL (Int #1)	1mL	20ug/mL	10uL	5ug/mL
8270-SIM Cal Std 8	250uL (Int #1)	1mL	50ug/mL	10uL	5ug/mL

9.2.3.12 Working 8270-SIM ICV Standard with Surrogates

Dilute 50uL of the Stock 8270-SIM ICV standard (200ug/mL) to 1mL with methylene chloride for a final concentration of 10ug/mL.

9.2.3.13 Working 8270-SIM Internal Standard

Dilute 375uL of the Stock Internal Standard (4000ug/mL) to3mL with methylene chloride for a final concentration of 500ug/mL.

9.2.3.14 Working 8270-SIM LVE Calibration Standards

The following are examples of calibration standards and could vary based on requirements:

Standard	8270 SIM Cal. Std. from Section 9.2.3.11	8270 SIM Cal. Std. Amount	Final Volume in Methylene Chloride	Final Calibration Standard Concentration	Internal Standard Concentration
8270-SIM LVE Cal Std 1	8270-SIM Cal Std 1	50uL	500uL	0.005ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 2	8270-SIM Cal Std 2	50uL	500uL	0.01ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 3	8270-SIM Cal Std 3	50uL	500uL	0.05ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 4	8270-SIM Cal Std 4	50uL	500uL	0.1ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 5 (CCV)	8270-SIM Cal Std 5	50uL	500uL	0.5ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 6	8270-SIM Cal Std 6	50uL	500uL	1.0ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 7	8270-SIM Cal Std 7	50uL	500uL	2.0ug/mL	0.5ug/mL
8270-SIM LVE Cal Std 8	8270-SIM Cal Std 8	50uL	500uL	5.0ug/mL	0.5ug/mL

9.2.3.15 Working 8270-SIM LVE ICV Standard with Surrogates

Dilute 50uL of the Working 8270-SIM ICV Standard with Surrogates (10ug/mL) to 500uL with methylene chloride for a final concentration of 1.0ug/mL.

9.2.3.16 Working 8270-SIM LVE Internal Standard

Dilute 37.5uL of the Stock Internal Standard (4000ug/mL) to3mL with methylene chloride for a final concentration of 50ug/mL.

10. Calibration

10.1. Calibration for 8270 Scan/8270 Scan LVE Analysis

10.1.1 8270 Scan DFTPP Tune Verification: At the beginning of each 8270 Scan analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer must be hardware tuned using a 50ng injection of DFTPP. Analysis must not begin until the tuning criteria are met. Use the DFTPP mass intensity criteria in the table below as tuning acceptance criteria. Alternate tuning criteria may be used provided that method performance is not adversely affected. The 12-hour window during which standards and samples may be analyzed begins with the injection of DFTPP. All subsequent standards, samples MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

Mass (m/z)	Ion Abundance criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

If the DFTPP ratios do not meet the criteria, reanalyze the DFTPP tune. If the DFTPP still fails the criteria, autotune adjustment, instrument maintenance, and/or preparation of new standards must be considered.

The mass spectrum of DFTPP may be acquired by averaging three scans, the peak apex scan and the scans immediately preceding and following the apex. Background subtraction is required using this approach and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak. Alternatively, the analyst may use other approaches suggested below:

1. A single scan within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.
2. An average of multiple scans within the DFTPP peak with background subtraction of a single scan no more than 20 scans prior to the elution of DFTPP.

10.1.2 Initial Calibration: Initial Calibration standards are introduced into the GC/MS from the lowest to highest concentration of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard as follows:

$$RF = \frac{(A_x)(C_{IS})}{(A_{IS})(C_x)}$$

where: A_x = Area of the quantitation ion for the compound being measured
A_{IS} = Area of the quantitation ion for the internal standard.
C_{IS} = Concentration of the internal standard
C_x = Concentration of the compound being measured.

10.1.3 The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

10.1.4 The percent relative standard deviation (%RSD) is calculated as follows:

$$\%RSD = \frac{(SD)}{RF_{avg}} \times 100$$

where: SD = Standard deviation of average RF for a compound
RF_{avg} = Mean of RFs for a compound

10.1.5 The %RSD should be should be $\leq$15% for each target analyte. However, the %RSD for each individual Calibration Check Compound (CCC) must be $\leq$30%. If the RSD of any CCC is >30%, then the chromatographic system is too reactive and instrument maintenance or preparation of new standards may be necessary before attempting recalibration. The CCCs are:

Base/Neutral Fraction	**Acid Fraction**
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

10.1.6 System Performance Check Compounds (SPCCs) are checked for a minimum average response factor (RF_{avg}) to determine potential instability and/or degradation caused by deterioration of instrument conditions or standard material. The minimum RF_{avg} for the semivolatile SPCCs are as follows:

N-nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Instrument maintenance or preparation of new standards may be necessary. The SPCC criteria must be met for sample analysis to begin.

10.1.7 If the percent relative standard deviation (%RSD) of the RFs for a compound is $\leq$15% over the calibration range, then linearity through the origin is assumed and the RF_{avg} may be used to determine sample concentrations.

10.1.8 If the % RSD for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where: y = instrument response (peak area)
a = slope of the line (the coefficient of x)
x = concentration of the calibration standard
b = intercept of the line

10.1.9 The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be $\geq$ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$r = \frac{\sum_{i=1}^{N} (X_i - \overline{X}) * (Y_i - \overline{Y})}{\sqrt{\left(\sum_{i=1}^{N} (X_i - \overline{X})^2\right) * \left(\sum_{i=1}^{N} (Y_i - \overline{Y})^2\right)}}$$

10.1.10 Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be $\geq$ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

$$\text{COD} = \frac{\sum_{i=1}^{N} (Y_i - \overline{Y})^2 - \left(\frac{n-1}{n-p}\right)\sum_{i=1}^{N} (Y_i - Y_i')^2}{\sum_{i=1}^{N} (Y_i - \overline{Y})^2}$$

10.1.11 Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards must also be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.1.12 Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the following equation:

$$\% \text{ Recovery} = \frac{\text{Observed concentration}}{\text{Theoretical concentration}} \times 100$$

The ICV is analyzed immediately following the initial calibration curve. The ICV recoveries are evaluated against a default acceptance range of 70-130% recovery.

10.1.13 **ICV Corrective Action:** If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

10.1.14 **Continuing Calibration Verification:** The initial calibration is verified every 12 hours by analyzing a DFTPP tune verification as described in Section 10.1, followed by a Continuing Calibration Verification (CCV) standard.

10.1.15 If the % difference (%D) or % Drift for each CCC is $\leq$20%, then the initial calibration is assumed to be valid. The response factors for all SPCCs in the CCV standard must meet the criteria in Section 10.6. **For BP, surrogate compounds must also be $\leq$20%D/Drift.**

$$\% \text{ Difference} = [RF_v - RF_{avg}] / RF_{avg} * 100$$

10.1.16 The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

10.1.17 **CCV Corrective Action:** If a CCV fails the acceptance criteria, check the instrument operating conditions and if necessary, restore them to the original settings, and analyze another CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

10.2 Calibration for 8270 SIM/8270-SIM LVE Analysis

10.2.1 The instrument is auto-tuned periodically according to manufacturer's instructions to maximize detector sensitivity. DFTPP tuning is not performed for this method. DFTPP tune data is acquired in scan mode. For SIM analysis, the instrument is set to selective ion monitoring (SIM) mode. Verification of hardware tuning is accomplished through the analysis of initial and continuing calibration standards in which the known ions are selected for data acquisition.

10.2.2 **Initial Calibration:** Initial Calibration standards are introduced into the GC from the lowest to highest concentration by direct injection of 1uL of each working calibration standard. The lowest calibration standard must be at or below the required reporting limit. Five calibration points, at a minimum, are analyzed to evaluate linearity. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the first calibration standard. Refer to the Quality Manual for more information regarding calibration curves. The response factor (RF) is calculated for each compound for each calibration standard using the calculation in Section 10.1.2.

10.2.3 The average response factor (RF_{avg}) is determined by averaging the response factors at the different concentrations for each target analyte

10.2.4 The percent relative standard deviation (%RSD) of the response factors is calculated using the calculation in Section 10.1.4.

10.2.5 All reported compounds in the calibration must be evaluated as CCC compounds. The %RSD of the RFs for each compound must be ≤30%.

10.2.6 All reported compounds in the calibration must be evaluated as SPCC compounds. Each compound must meet a minimum average response factor of 0.050.

10.2.7 If the %RSD of the RFs for any compound is >15%, the analyst may employ a regression equation that does not pass through the origin.

10.2.8 The regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be ≥ 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

10.2.9 Non-linear or quadratic calibration: A non-linear or quadratic calibration model can only be used if the compound(s) have historically exhibited a non-linear response and cannot be used to extend the calibration range for any compound that normally exhibits a linear response in a narrower range. The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of: $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. Because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. The "goodness of fit" of the polynomial equation is evaluated by calculating the coefficient of the determination (COD) or r^2. The COD or r^2 from the regression equation must be greater than or equal to 0.99. Refer to SW-846 Method 8000C for detailed information regarding equations.

10.2.10 Initial Calibration Corrective Action: If the initial calibration curve does not meet the required criteria to be used for quantitative purposes, a new initial calibration curve must be analyzed. Instrument maintenance and/or preparation of new calibration standards should also be considered. Samples associated with a failed initial calibration must be reanalyzed.

10.2.11 Initial Calibration Verification (ICV): In addition to meeting the response and linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known true value. This step is referred to as the Initial Calibration Verification. The ICV must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. The accuracy of the standard is assessed as a percent recovery (%Rec) of the observed ICV according to the equation in Section 10.1.12.

10.2.12 ICV Corrective Action: If the ICV exceeds the acceptance range, another ICV may be analyzed. If the second ICV also exceeds the acceptance range, a new initial calibration should be prepared. Alternatively, the allowance for use of the ICV when the criterion is not met is documented and is at the discretion of the Department Manager, Quality Manager, General Manager or designee.

10.2.13 Continuing Calibration Verification: The initial calibration is verified every 12 hours by analyzing a Continuing Calibration Verification (CCV) standard. The CCV is prepared using the same standard solution used for the initial calibration. The 12 hour clock during which standards and samples may be analyzed begins with the injection of the CCV.

10.2.14 If the % Difference (%D) or % Drift for each CCC is ≤20%, then the initial calibration is assumed to be valid. Each SPCC must meet the SPCC criteria in Section 10.2.6. All reported compounds are considered CCCs and SPCCs for the evaluation of the CCV. **For BP, surrogate compounds must also be ≤20%D/Drift.**

10.2.15 The internal standard areas in the CCV must be between 50%-200% of the internal standard areas of the corresponding standard in the initial calibration. In addition, the retention time of the internal standards in the CCV cannot shift by more than 30 seconds from the corresponding standard in the initial calibration. Failure in either of these two areas requires the analyst to evaluate their system and perform maintenance if necessary.

10.2.16 CCV Corrective Action: If a CCV fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the CCV. If the response still fails the acceptance criteria, then a new initial calibration must be prepared. Samples associated with a failed CCV must be reanalyzed. **Exception:** If the CCV is outside of the upper control limit, indicating high bias, associated samples determined to be <RL may be reported.

11 Procedures

11.1 All sample extracts must be analyzed at room temperature and the instrument systems must be tuned, if required, calibrated as per Section 10, and free of contamination before samples are analyzed.

11.2 Gas Chromatography conditions: Configure the GC/MS per manufacturer's instructions.

11.3 For 8270 Scan/8270-SIM extracts: The extract obtained from the sample preparation laboratory is divided equally into two separate vials. The vial for 8270 Scan analysis should be fortified with 5uL of the Stock 8270 internal standard (4000ug/mL) just prior to analysis, resulting in an internal standard concentration of 40ug/mL. The vial for 8270-SIM analysis should be fortified with 5uL of the working SIM internal standard solution (500 ug/mL) just prior to analysis, resulting in an internal standard concentration of 5ug/mL. Analyze each extract by injecting 1uL.

11.4 For 8270 Scan LVE/8270-SIM LVE extracts: The extract obtained from the sample preparation laboratory is divided equally into two separate vials. The vial for 8270 Scan LVE analysis should be fortified with 5uL of the Working 8270 LVE internal standard (400ug/mL) just prior to analysis, resulting in an internal standard concentration of 4ug/mL Analyze each 8270 Scan LVE extract by injecting 10uL. The vial for 8270-SIM LVE analysis should be fortified with 5uL of the Working 8270-SIM LVE internal standard (50ug/mL) just prior to analysis, resulting in an internal standard concentration of 0.5ug/mL. Analyze each 8270-SIM LVE by injecting 1uL.

11.5 Qualitative Analysis

11.5.1 The relative retention time (RRT) of the sample component must compare within +/- 0.06 RRT units of the RRT of the CCV component.

11.5.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Refer to Table 2 for characteristic ions.

11.5.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.6 Quantitative analysis: Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique. See Sections 11.7, 11.8, and 11.9 for equations to calculate the amount or analyte or surrogate introduced into the instrument. Calculations are subject to

change based on the data reduction software used. Refer to the current Manual Integration SOP for manual integration guidelines.

11.7 When the average response factor from the initial calibration is used, calculate the on-column amount:

$$X_s = \left(\frac{A_s}{RF} \right) x \left(\frac{C_{is}}{A_{is}} \right)$$

11.8 When a linear curve fit is used, calculate the on-column amount:

$$X_s = \left[b + \frac{\left(\frac{A_s}{A_{is}} \right)}{m1} \right] * C_{is}$$

11.9 When a non-linear or quadratic curve fit is used, calculate the on-column amount:

$$X_s = \left[b + m1\left(\frac{A_s}{A_{is}} \right) + m2\left(\frac{A_s}{A_{is}} \right)^2 \right] * C_{is}$$

Where:
RF = Average Response Factor
X_s = On-column concentration of the analyte or surrogate
A_s = Response of the analyte or surrogate in the sample
A_{is} = Response of the internal standard in the sample
C_{is} = Concentration of the internal standard in the sample
m1 = Slope of the line
b = The y-intercept
m2 = coefficient of the determination

11.10 Calculate the final concentration in the sample as follows:

Aqueous Sample (ug/L) = $\frac{(X_s)(V_t)(D)}{(V_i)(V_s)}$ Solid Sample (ug/Kg) = $\frac{(X_s)(V_t)(D)}{(V_i)(W_s)}$

Where:
X_s = On-column concentration of the analyte in the sample aliquot injected
V_t = Total volume of concentrated extract
D = Dilution factor
V_i = Volume of the extract injected in uL
V_s = Volume of aqueous sample extracted in milliliters
W_s = Weight of solid sample extracted in grams

Moisture corrected concentration = $\frac{\text{(Final concentration as received)}}{\text{(100 - \%Moisture)}} \times 100$

12 Quality Control

12.1 Batch Quality Control

Table 12.1 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Reagent water	One per preparation batch of up to 20 samples, per matrix.	Target analytes must be less than reporting limits.	Re-extract and re-analyze if target compound is >RL in method blank and associated samples. *Exceptions:* 1) If the method blank concentration is less than 1/10 of the amount measured in the sample, corrective action is not required, affected data must be qualified. 2) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, the reported method blank and samples must be qualified. 3) If a contaminant is present only in the method blank and not the samples, no action is required.
Laboratory Control Sample (LCS)	Applicable target analytes	One per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits. Refer to Sections 12.1.1 and 12.1.2 for additional information.	Re-extract and re-analyze associated samples if original LCS is outside acceptance limits. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified. 2) If LCS recovery is >QC limits and sample results are non-detect, the sample data may be reported. The LCS data must be qualified.
Matrix Spike (MS)/Matrix Spike Duplicate (MSD)	Applicable target analytes	One MS/MSD set per preparation batch of up to 20 samples, per matrix.	Lab-generated limits Refer to the LIMS for acceptance limits.	No corrective actions necessary. If LCS recovery is in range, the system is considered in-control and the out-of-control MS/MSD must be qualified appropriately.
Surrogates	Applicable surrogate compounds	Added to each standard, sample, and method blank.	Lab-generated limits Refer to the LIMS for acceptance limits.	Samples with surrogate failures must be re-extracted and reanalyzed. *Exceptions:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported surrogate data must be qualified. 2) If surrogate result is >QC limits, and sample or method blank results are non-detect, the sample or method blank results may be reported. The surrogate must be qualified. 3) If only one surrogate fails and it is >10% recovery, re-extraction is not required but data must be qualified. 4) MS/MSD surrogate recovery failures do not constitute the re-extraction or reanalysis of samples but the surrogate data must be qualified.
For BP only: **Internal Standards**	Applicable Internal Standard compounds	Added to each standard, sample, and method blank.	Sample ISTD areas must be -50% to +100% from CCV. Sample ISTD RTs must be +/-0.5 minutes from CCV.	Samples with internal standard failures must be reanalyzed undiluted or more concentrated. The laboratory may only dilute a sample prior to reanalysis if matrix interference is present. *Exception:* 1) If no additional sample remains for reanalysis or if reanalysis cannot take place within holding time, reported data must be qualified.

12.1.1 The matrix spike may be used in place of the LCS as long as the acceptance criteria are as stringent as for the LCS.

12.1.2 **Allowable Marginal Exceedances**: If a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside control limits. A marginal exceedance (ME) is defined as being beyond the LCS control limit of +/-3 standard deviations, but within the ME limits of +/-4 standard deviations around the mean. The number of allowable MEs is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and correction action is necessary. If the same analyte exceeds the LCS control limit consecutively, it is an indication of a systemic problem. The source of the error shall be located and corrective action taken.

The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number Allowed as Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

NOTE: For BP, the LCS shall be allowed to be outside the control limits but ≥10% for hexachlorocyclopentadiene, N-nitrosodimethylamine, pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, and 4-chloroaniline without corrective action. The LCS shall be allowed to be outside the control limits but >10% for up to four additional compounds, with the exception of any PAH, without corrective action. All PAH compounds must be within control limits.

12.2 LCS equation

$$R = (C/S) * 100$$

Where R = percent recovery
C = observed LCS concentration
S = concentration of analyte added to the clean matrix

12.3 MS/MSD equation

$$R = \frac{(Cs - C)}{S} * 100$$

Where R = percent recovery
Cs = observed spiked sample concentration
C = sample concentration
S = concentration of analyte added to the sample

12.4 RPD calculations:

$$RPD = \frac{|D_1 - D_2|}{[(D_1 + D_2)/2]} * 100$$

Where RPD = relative percent difference
D_1 = first sample result
D_2 = second sample result

13 Method Performance

13.1 **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually for 8270 Scan and 8270 SIM per instrument.

13.2 **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) for 8270 Scan and 8270 SIM.

14 Method Modifications

14.1 Standards are purchased as certified stock solutions and not prepared from neat materials.

14.2 Phenol-d5 is used as a surrogate instead of Phenol-d6.

14.3 Extract final volumes, volume of internal standards added to extracts, and volume of extract injected into the instrument may vary from those identified in Method 8270C.

15 Pollution Prevention and Waste Management

15.1 Procedures for handling waste generated during this analysis are addressed in S-IN-S-002, Waste Handling, or other applicable SOP. All wastes are accumulated, managed and disposed of in accordance with all federal and state laws and regulations.

15.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

15.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

16 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B, 8000C (for detailed calibration equations only) and 8270C

16.2 Pace Manual Integration SOP, latest revision.

16.3 Pace Analytical Quality Manual; latest revision.

16.4 TNI Standard; Quality Systems section; latest revision.

17 Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

17.1 Table 1: 8270 Scan/8270 Scan LVE Target Compounds and Reporting Limits

17.2 Table 2: 8270 Scan/8270 Scan LVE Characteristic Ions of Target Compounds

17.3 Table 3: 8270 SIM/8270 SIM LVE Target Compounds and Reporting Limits

17.4 Table 4: 8270 SIM/8270 SIM LVE Characteristic Ions of Target Compounds

18 Revisions

Document Number	Reason for Change	Date
S-IN-O-163-rev.00	1. First Issue	07Apr2011
S-IN-O-163-rev.01	1. Table of Contents: added new Section 14, Method Modifications 2. Section 2.2: removed reference to soil extraction. 3. Section 3.1: added reference to MDLs 4. Table 7.1: added reference to LVE container, removed reference to solid samples 5. Table 8.1: added reference to SIM mode for MS 6. Table 8.2: added column detail for SIM instrument 7. Table 9.2: clarified that DFTPP tune is not performed for SIM analysis 8. Table 9.3a: revised for standards currently used and added LVE standards 9. Table 9.3b: added LVE standards 10. Section 9.2.3: revised for standards currently used and added LVE standards for both 8270 Scan and 8270 SIM. 11. Sections 10.1.12 and 10.1.13: revised ICV language to reflect actual practice. 12. Sections 10.2.11 and 10.2.12: revised ICV language to reflect actual practice. 13. Section 11.4: added ISTD instructions and injection volume for LVE extracts. 14. Section 11.6: added reference to Manual Integration SOP for guidance. 15. Sections 11.7-11.9: replaced 8000C calculations with Target calculations. 16. Table 12.1: revised corrective action for Method Blank 17. New Section 14, Method Modifications added. 18. Section 15.1: updated SOP reference 19. Section 16: added reference to Manual Integration SOP and changed NELAC to TNI 20. Section 17: added LVE references to table titles. 21. Table 1 and Table 3: removed references to soil RLs.	16May2013

Table 1: 8270 Scan/8270 Scan LVE Target Compounds and Reporting Limits[1]

Analyte	RL water (ug/L)
Phenol	10
Bis (2-chloroethyl) ether	10
2-Chlorophenol	10
Benzyl Alcohol	20
2-Methylphenol (o-Cresol)	10
Bis (2-chloroisopropyl)ether	5
3&4-Methylphenol (m&p-Cresol)	20
N-Nitroso-di-n-propylamine	10
Hexachloroethane	10
Nitrobenzene	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
Bis(2-chloroethoxy)methane	10
2,4-Dichlorophenol	10
Naphthalene	5
4-Chloroaniline	20
Hexachlorobutadiene	5
4-Chloro-3-methylphenol	20
1-Methylnaphthalene	10
2-Methylnaphthalene	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2,4,5-Trichlorophenol	10
2-Chloronaphthalene	10
2-Nitroaniline	50
Dimethyl phthalate	10
Acenaphthene	10
Acenaphthylene	10
2,4-Dinitrophenol	50
4-Nitrophenol	50
Dibenzofuran	10
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
3-Nitroaniline	50
Diethyl phthalate	10
4-Chlorophenyl phenyl ether	10
Fluorene	10
4-Nitroaniline	50
4,6-Dinitro-2-methylphenol	50
N-Nitrosodiphenylamine	10
4-Bromophenyl phenyl ether	10

[1] Target Compounds and Reporting Limits are subject to change.

Analyte	RL water (ug/L)
Hexachlorobenzene	10
Pentachlorophenol	50
Phenanthrene	10
Anthracene	10
Di-n-butyl phthalate	10
Fluoranthene	10
Pyrene	10
Butyl benzyl phthalate	10
3,3'-Dichlorobenzidine	20
Benzo(a)anthracene	10
Chysene	10
Bis(2-ethylhexyl)phthalate	5
Di-n-octyl phthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(a)pyrene	10
Indeno(1,2,3-cd)pyrene	10
Dibenz(a,h)anthracene	10
Benzo(g,h,i)perylene	10

[1] Target Compounds and Reporting Limits are subject to change.

Table 2: 8270 Scan/8270 Scan LVE Characteristic Ions of Target Compounds[2]

Analyte	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
Bis (2-chloroethyl) ether	93	63, 95
2-Chlorophenol	128	64, 130
Benzyl Alcohol	108	77, 79
2-Methylphenol (o-Cresol)	107	77, 108
Bis (2-chloroisopropyl)ether	45	77, 121
3&4-Methylphenol (m&p-Cresol)	107	77, 108
N-Nitroso-di-n-propylamine	70	42, 101
Hexachloroethane	117	199, 201
Nitrobenzene	77	65, 123
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	122	107, 121
Bis(2-chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	98, 164
Naphthalene	128	127, 129
4-Chloroaniline	127	65, 129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	142, 144
1-Methylnaphthalene	142	141
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	165, 194
Acenaphthene	154	152, 153
Acenaphthylene	152	151, 153
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	139	65, 109
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
3-Nitroaniline	138	92, 108
Diethyl phthalate	149	150, 177
4-Chlorophenyl phenyl ether	204	141, 206
Fluorene	166	165, 167
4-Nitroaniline	138	65, 108
4,6-Dinitro-2-methylphenol	198	51, 105
N-Nitrosodiphenylamine	169	167, 168
4-Bromophenyl phenyl ether	248	141, 250

[1] Target Compounds and Reporting Limits are subject to change.

Analyte	Primary Ion	Secondary Ion(s)
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	176, 179
Anthracene	178	176, 179
Di-n-butyl phthalate	149	104, 150
Fluoranthene	202	101, 203
Pyrene	202	200, 203
Butyl benzyl phthalate	149	91, 206
3,3'-Dichlorobenzidine	252	126, 254
Benzo(a)anthracene	228	226, 229
Chysene	228	226, 229
Bis(2-ethylhexyl)phthalate	149	167, 279
Di-n-octyl phthalate	149	43, 167
Benzo(b)fluoranthene	252	125, 253
Benzo(k)fluoranthene	252	125, 253
Benzo(a)pyrene	252	125, 253
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Internal Standards	**Primary Ion**	**Secondary Ion(s)**
1,4-Dichlorobenzene-d4	152	150, 115
Naphthalene-d8	136	68
Acenaphthene-d10	164	160, 162
Phenanthrene-d10	188	80, 94
Chrysene-d12	240	120, 236
Perylene-d12	264	260, 265
Surrogates	**Primary Ion**	**Secondary Ion(s)**
Phenol-d5	99	42, 71
2-Fluorophenol	112	64, 77
Nitrobenzene-d5	82	54, 128
2-Fluorobiphenyl	172	171
2,4,6-Tribromophenol	330	141, 332
Terphenyl-d14	244	122, 212

[2] Target Compounds and primary ions are subject to change.

Table 3: 8270 SIM/8270 SIM LVE Target Compounds and Reporting Limits[1]

Analyte	RL aqueous (ug/L) 8270 SIM
Acenaphthene	1
Acenaphthylene	1
Anthracene	0.1
Benzo(a)anthracene	0.1
Benzo(a)pyrene	0.1
Benzo(b)fluoranthene	0.1
Benzo(g,h,i)perylene	0.1
Benzo(k)fluoranthene	0.1
Chrysene	0.5
Dibenz(a,h)anthracene	0.1
Fluoranthene	1
Fluorene	1
Indeno(1,2,3-cd)pyrene	0.1
1-Methylnaphthalene	1
2-Methylnaphthalene	1
Naphthalene	1
Phenanthrene	1
Pyrene	1

[1] Target Compounds and Reporting Limits are subject to change.

Table 4: 8270 SIM/8270 SIM LVE Characteristic Ions of Target Compounds[2]

Analytes	Primary Ion	Secondary Ion
Acenaphthene	154	153
Acenaphthylene	152	151
Anthracene	178	176
Benzo(a)anthracene	228	229
Benzo(a)pyrene	252	253
Benzo(b)fluoranthene	252	253
Benzo(g,h,i)perylene	276	138
Benzo(k)fluoranthene	252	253
Chysene	228	226
Dibenz(a,h)anthracene	278	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	200
Internal Standards	**Primary Ion**	**Secondary Ion**
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162
Phenanthrene-d_{10}	188	94
Chrysene-d_{12}	240	120
Perylene-d_{12}	264	260
Surrogates	**Primary Ion**	**Secondary Ion**
2-Fluorobiphenyl	172	171
Terphenyl-d_{14}	244	122

[2] Target Compounds and primary ions are subject to change.

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STANDARD OPERATING PROCEDURE

DETERMINATION OF HEXAVALENT CHROMIUM

Reference Methods: EPA Method 218.6 and 218.7

SOP Number:	S-FL-I-050-rev.07
Effective Date:	Date of Final Signature
Supersedes:	S-FL-I-050-rev.06

APPROVALS

Bob Dempsey,
General Manager — Date: 6-1-15

Tina Buttermore,
Quality Manager — Date: 5/28/15

Trisha Kelly,
Ormond Department Supervisor — Date: 6/1/15

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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1. Purpose

This SOP documents the steps and conditions involved in the analysis of Hexavalent Chromium (Cr+6) by Ion Chromatography (IC) using EPA method 218.6. Additional criteria is also included for EPA 218.7 in support of UCMR 3.

2. Scope and Application

2.1. This method is applicable to drinking water, ground water and industrial effluents.

2.2. The method reporting limit for this application is 0.025ug/L (25ng/L). The reporting limit for UCMR3 is 0.03 ug/L.

2.3. In accordance with SOP S-ALL-Q-004 Determination of Limit of Detection (LODs) and Limit of Quantitation (LOQs) or its equivalent revision or replacement, Method Detection Limits (MDLs) are performed on an on-going basis. Method Detection Limits are listed in the Laboratory Information Management System (LIMS).

2.4. This method is restricted to use by or under the supervision of analysts experienced in the use of ion chromatography equipment. Each analyst must demonstrate the ability to generate acceptable results with this method.

3. Summary of Method

3.1. A small volume of sample, typically 25-250µL, is introduced into an ion chromatograph. A guard column removes the organics from the sample before the Cr+6 is separated on a high capacity anion exchange separator column. Post –Column derivatization of the Cr+6 with diphenylcarbazide is followed by detection of the colored complex at 530nm.

3.2. Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in the Quality Control section.

4. Interferences

4.1. Interference can be caused by samples containing large amounts of anioinic species such as sulfate and chloride. Samples containing large amounts of sulfide may cause rapid reduction of Cr+6 to Cr+3.

4.2. Contamination may occur from improperly cleaned glassware or contact of caustic or acidic reagents or samples with stainless steel or pigmented material.

5. Safety

5.1. Precautions should be taken while handling all chemicals and while working on any instrumentation. Each chemical compound and sample should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. It is mandatory to wear personal safety equipment while working with chemicals and samples. A reference file of material data handling sheets (MSDS) is available for review in the laboratory. Use judgment and good laboratory procedures while working in the laboratory. Please direct any questions to your direct supervisor. Pace Analytical Services-Florida Chemical Hygiene Plan provides overall guidance on laboratory safety protocols.

5.2. Hexavalent Chromium is a toxic known carcinogen and should be handled carefully. Analysts should be careful when weighing the salt for preparation of the standard.

6. Definitions

6.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

6.2. Linear Dynamic Range (LDR): The concentration range over which the instrument response is linear. The LDR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. This study will be performed as follows:

6.2.1. A blank and the top three calibration standards of the standard calibration curve are analyzed against an acceptable, current calibration.

6.2.2. An additional standard(s), at concentrations above the highest calibration standard, is (are) analyzed. Their observed concentrations are compared to their known values.

6.2.3. The LDR is the concentration range of those standards who have observed concentrations varying by less than 10% from their known values. The standard concentration at which the observed concentration does not meet the 10% criteria is that point that is defined as outside of the LDR.

6.2.4. Any sample exceeding the highest standard of the curve will be diluted so that the concentration falls within the calibration standards of the curve.

7. Sample Collection, Preservation, and Handling

7.1. Laboratory samples are collected, preserved, shipped, and stored in accordance with Pace Analytical Services-Florida SOP S-ALL-C-004 Bottle Preparation.

7.2. Refer to F-FL-C-039 most current revision or replacement, for the preservation requirements including special projects.

7.3. For ESI BP-XA, samples must be preserved in the laboratory with Adjustment Buffer 2 (section 9.5) to a pH = 9.3-9.7 within 7 days of collection. After preservation the hold time is 14 days from collection.

7.3.1. Upon receipt of samples, filter with 0.45um pore size filters, and preserve and adjust pH to 9.3-9.7 using the Ammonium Adjustment Buffer 2.

7.3.2. Filter MB and LCS with 0.45um pore size filter before running.

7.4. For dissolved Cr+6 by 218.6, filter the sample through a 0.45um filter prior to analysis. Field filtration is recommended.

7.5. In order to protect the instrument from samples containing suspended solids, samples can be filtered with a 2.0um filter, per analyst discretion.

7.6. The holding time for all unpreserved samples is 24 hours.

8. Equipment and Supplies

8.1. Dionex ICS-1100 or equivalent.

8.2. Ion Pac AS-7 column or equivalent.

8.3. IonPac AG-7 guard column or equivalent.

8.4. 1000µL loop or equivalent.

8.5. 375uL reaction coil or equivalent.

8.6. VWD UV-VIS absorbance detector or equivalent.

8.7. PC10/AXP post-column pneumatic delivery package, or equivalent.

9. Reagents and Standards

9.1. Pace Analytical Services-Florida SOP S-ALL-Q-025 Standard and Reagent Prep and Traceability (or its equivalent revision or replacement) provides guidance on the laboratory's process for handling chemicals, reagents, and standards.

9.2. **Eluent-** Dissolve 66g of ammonium sulfate in ~1L of DI water and add 13mL of 29% ammonium hydroxide solution. Dilute to 2.0L with DI water.

9.3. **Post Column Reagent**- Add 28mL of 98% sulfuric acid to ~500mL of DI water in a 1.0L volumetric flask (caution: this mixture may get hot). Mix and allow to cool. Add 0.5g of 1,5-diphenylcarbazide to ~75mL of HPLC-grade methanol in a 100mL volumetric flask and sonicate to dissolve. Bring to volume with methanol, mix, and add to the cooled sulfuric acid solution. Dilute to 1.0 L with DI water, mix, and transfer to the pressurized PCR container. Prepare fresh as needed. **Note**: The PCR is stable for a maximum of 2 days, so it is recommended that only 500mL of reagent be made at one time to eliminate excess waste.

9.4. **Adjustment Buffer-1 (218.7) (250mM Ammonium sulfate + 1000mM Ammonium hydroxide) -** Dissolve 3.3g of ammonium sulfate in ~75mL of DI water and add 6.5mL of 29% ammonium hydroxide. Dilute to 100mL with DI water. This buffer should be used for DW and UCMR samples, and calibration and QC standards.

9.5. **Adjustment Buffer-2 (218.6)** Dissolve 33g of Ammonium sulfate in ~75mL of DI water and add 6.5mL of 29% ammonium hydroxide. Dilute to 100mL with DI water. This buffer should be used for ESI BP-XA and non-potable samples.

9.6. **Chromium Stock standard 10000mg/L**- Dissolve 0.238g of Potassium Dichromate ($K_2Cr_2O_7$) which has been dried at 100°C for 1 hour into a 100mL volumetric flask. Bring to volume with DI water. **Note: glassware for Hexavalent Chromium should be segregated from general wet chem glassware.** This standard should be stored at 4 +/-2 °C up to 6 months.

9.7. **1mg/L Chromium intermediate standard**- Volumetrically dilute 0.1mL of 1000mg/L Chromium Stock standard into 100mL of DI water. Make fresh weekly.

9.8. **10ug/L Chromium intermediate standard-** Volumetrically dilute 1mL of 1mg/L Chromium intermediate into 100mL DI water. Make fresh weekly.

9.9. **Calibration Standards:** all made from 10ug/L Chromium intermediate standard. Volumetrically dilute the following amounts into 100mL DI. Expires 24hr after preparation.

True Value Working Chromium Standard	Amount of 10ug/L Intermediate added	Amount of Adj. Buffer 1 added	Final Volume
300ng/L Cr+6	3.00mL	1mL	100mL
250ng/L Cr+6	2.50mL	1mL	100mL
200ng/L Cr+6	2.00mL	1mL	100mL
150ng/L Cr+6	1.50mL	1mL	100mL
125ng/L Cr+6	1.25mL	1mL	100mL
100ng/L Cr+6	1.00mL	1mL	100mL
75ng/L Cr+6	750uL	1mL	100mL
50ng/L Cr+6	500uL	1mL	100mL
25ng/L Cr+6	250uL	1mL	100mL

9.10. **Second Source Chromium Stock Standard 1000mg/L -** Dry Potassium Chromate at 100°C for 1 hour and to a constant weight. Constant weight is achieved when there is no change in weight after two consecutive drying periods of no less than 1 hour. Dissolve 0.3738g of dried Potassium Chromate (K_2CrO_7) into a 100mL volumetric flask. Bring to volume with DI water. This standard should be stored at 4ºC up to 6 months. **Note: glassware for Hexavalent Chromium should be segregated from general wet chem glassware.**

9.11. **1mg/L Second Source Intermediate Standard**- Volumetrically dilute 0.1mL of the 1000mg/L Second Source Chromium Stock Standard into 100mL of DI water. Make fresh weekly.

9.12. **10ug/L Second Source Intermediate Standard-** Volumetrically dilute 1mL of the 1mg/L Second Source Chromium Intermediate Standard into 100mL DI water. Make fresh weekly.

9.13. **Second Source Working Standards**- all prepared from the 10ug/L Second Source Intermediate Standard. Make fresh daily.

QC Standard	True Value Working Chromium Standard	Amount of Second Source Intermediate added	Amount of Adj. Buffer 1 added	Final Volume
CCV-High	200ng/L Cr+6	2.00mL	1mL	100mL
CCV-Mid / LCS	75ng/L Cr+6	0.75mL	1mL	100mL
RLVS	25ng/L Cr+6	0.25mL	1mL	100mL

9.14. **Matrix spikes:** alternate between the Low and High MS/MSD. Refer to Table 1 for frequency and acceptable limits.

QC Standard	True Value	Amount of 10ug/L Second Source Intermediate added	Final Volume
MS/MSD High	75ng/L Cr+6	75.0uL	10.0mL
MS/MSD Low	25ng/L Cr+6	25.0uL	10.0mL

9.15. **Method Blank (MB / LRB /CCB):** prepare by diluting 1.0mL of Adjustment Buffer-1 into 100mL DI water.

10. Calibration

10.1. Refer to Pace Analytical Services – Florida SOP S-ALL-Q-013 for additional information on calibration requirements for support equipment that may be used in this procedure.

10.2. Instrument Set up: the following are suggested settings:

10.2.1. ICS-1100 Pump Setup.

10.2.2. Pump Flow rate – 0.36mL/minute.

10.2.3. System back pressure- 1000-3000psi.

10.3. Detector Setup: the following are suggested settings:

10.3.1. 530nm VIS (required wavelength), Tungsten lamp.

10.3.2. Data collection rate- 2.5 Hz.

10.3.3. Time constant – 1.0 second.

10.4. AXP pump: the following are suggested settings:

10.4.1. Flow rate: 0.12-0.14mL/min.

10.4.2. Helium head pressure- 2 psi.

10.4.3. Configure AXP pump as described in AXP/AXP-MS manual. Use green peek tubing between the AXP pump and backpressure tubing. Connect using a union to reduce pump noise. A well equilibrated system should have peak to peak noise less than 10uAU.

10.5. Instrument Shutdown Short term (as necessary):

10.5.1. After running a sequence of injections, if the system will not be running this application for a few days, it is recommended to run the pump used for PCR delivery with 1:1 Methanol. The VIS lamp and column heater should be turned off to extend its life.

10.5.2. Turn off AXP pump from the front control panel, and turn off the PC10 pump by turning the tab to "Vent."

10.5.3. Replace the PCR reagent with 1:1 Methanol.

10.5.4. Turn the AXP and PC10 pumps back on and flow 1:1 Methanol (rinsing down) through the system. The total flow rate is ~0.5mL per minute, so be sure to have enough methanol in both reagent bottles until return.

10.6. Instrument Shutdown (Long term, shutdown for >1 week, as needed per analyst discretion): When the system is idle for longer periods, the pump can be run with DI water at a reduced flow rate to achieve rapid startup.

10.6.1. Turn AXP pump off from front control panel.

10.6.2. Turn the ICS-1100 pump off from Chromeleon control panel tabset.

10.6.3. Prepare the guard/column set for storage by removing the guard and column from the system and plug the ends to keep the resin from drying out.

10.6.4. In place of the column, a length of backpressure tubing yellow PEEK tubing (P/N 052301 or equivalent): couple the column inlet and column outlet lines with a PEEK union and a length of back pressure tubing.

10.6.5. Replace both the eluent and PCR with DI WATER and flush the system thoroughly (ICS-1100 pump flow at 1.00mL/min; AXP flow at 1.00mL/min) for about 2 hours, periodically switching the injection valve between load and inject (using the toggle button in the CM panel tabset).

10.6.6. Once the above steps are completed, turn off both pumps and the VWD lamp; AXP shut off from the front control display panel and the IC from the Chromeleon control panel tabset.

11. Procedure

11.1. Starting the computer:

11.1.1. Click on the Chromeleon icon on the desk top. The software will open to the browser window.

11.1.2. Click on the default panel tabset icon. Select my computer, Chromeleon server, and the OK. This will bring up the panel to the ICS 1100 tab.

11.1.3. On the ICS 1100 tab, click on the pump settings button, and turn the pump on. Close the window. Then click on the heater settings, turn the temperature to 30.1, hit enter, and then close the window.

11.1.4. Click on the VWD tab and turn the Vis lamp on by clicking on the box. The light will turn green when it is lit.

11.1.5. On the PC10, turn the switch from Vent to On, and on the AXP, press the Run/Stop button to begin the Post Column flow.

11.1.6. On the panel, select the Sequence Control tab and click on Acq On to begin acquiring data to view the baseline. After about 10 minutes, click the Acq Off button.

11.2. Creating a Sequence:

11.2.1. To create a new sequence from scratch, select 'file', 'new', 'sequence' (using wizard), and 'OK'.

11.2.2. Select the '+' next to 'My Computer', then select the ICS-1100, and then click 'next'.

11.2.3. Enter the number of sample vials needed, along with the start position, and then click 'apply' followed by 'next'.

11.2.4. Enter the number of standard vials needed, along with the start position, and then click 'apply' followed by 'next'.

11.2.5. Next to the preferred report, select 'browse' and then click on 'default' and then 'open'.

11.2.6. Name the sequence and then click 'finish', followed by 'done'.

11.2.7. To create a sequence from a current working calibration, open a sequence with the current calibration.

11.2.8. Select "File/Save As". From the window that pops up, check "Save Raw Data", and rename to the current date name, following the format 35WTAB-YYYYMMDD.

11.2.9. When the new sequence opens, delete the old sample IDs, and type the sequence below the calibration standards. Make sure the "Status" is set to "single".

11.3. Creating a Program, if needed:

11.3.1. Highlight the sequence you wish to add the program to, and then select 'file', 'new', 'program file', and then 'OK'.

11.3.2. Select the '+' next to 'My Computer', then select the ICS-1100, and then click 'next'.

11.3.3. Make sure the flow is set to 0.36 and click 'next', and then 'next' again.

11.3.4. Change the acquisition time from 0-30 to 0-10 and click 'next'.

11.3.5. Change the acquisition on time from 0.000 to 0.100, and change the time constant to 1. Then click 'next', and 'next' again.

11.3.6. Name the program file, select 'review program', and then click 'finish'.

11.3.7. When the program file opens, review the new program. If everything is accurate, select 'file', 'save as', and 'save'.

11.3.8. In the sequence, click on the first box under the program column and click on the down arrow. Select the new program and then press F9 to populate it for the entire sequence.

11.4. Creating a Method, if needed:

11.4.1. Highlight the sequence you wish to add the method to, and then select 'file', 'new', 'method file', and 'OK'.

11.4.2. Click on the general tab at the bottom of the screen, and type the method name in the title box.

11.4.3. Type 'ng/L' in the dimensions box.

11.4.4. Select 'file', 'save as', and 'save'.

11.4.5. In the sequence, click on the first box under the method column and click on the down arrow. Select the new method and then press F9 to populate it for the entire sequence. Note: The program is what runs the instrument while the method is what quantifies the data. Once a sequence is created with a functioning program and suitable method, it is sufficient to select an old sequence and click 'file' and save it as a new sequence so that the above steps do not need to be repeated each time a run is performed.

11.5. Running a Sequence:

11.5.1. If needed, start up the instrument and wait until it has stabilized. It is recommended that the flow rate be checked before each run.

11.5.2. Load all samples in the autosampler. Note: When the autosampler finishes a sequence, the needle remains inside the last cup. In order to get the needle out so that the loading tray may be spun around, the carousel release button on the left side of the module under the lid must be pressed. Afterwards, ensure the autosampler has been reset in the software.

11.5.3. From the browser window, select the sequence you wish to run. At the top of the window, click on 'batch' and select 'start'.

11.5.4. Select the 'ready check' button and review the message stating the amount of disk space and reagent volume necessary to complete the run. Make sure there is a sufficient reagent in the carboys, and then click 'OK'.

11.5.5. Select 'start', and within seconds the first line in the sequence should become highlighted green to ensure that the sample is running.

11.6. Quantifying Data (New Methods Only):

11.6.1. From the browser window, select the current sequence and double click on any standard that has finished running.

11.6.2. Click on the 'QNT editor' button, and then select the detection tab at the bottom of the window. Make sure that the box under 'parameter name' reads 'minimum area', and double click the box under parameter value. Change the signal to 0.01 to eliminate the unnecessary integration of multiple peaks.

11.6.3. Next, select the peak table tab and right click anywhere in the grey area. Select 'autogenerate peak table', and then click on 'enumerate peaks' followed by 'OK' twice.

11.6.4. Change the name of the first peak to 'unknown' and name the second peak Cr+6.

11.6.5. Double click the box under cal type for the Cr+6 peak, and uncheck 'force through zero'. Then check on 'add zero point for curve fitting'.

11.6.6. Select the amount table tab and right click anywhere in the grey area. Select 'columns, and then edit amount columns. Assign the standard on the basis of name, click 'autogenerate', and click 'apply, and 'OK'. Manually change the amounts for the Cr+6 peak under each column on the amount table tab. Press 'save'.

11.6.7. Once all the standards in the sequence have run, click on any standard and click on the 'integration window' button. Select the calibration tab at the bottom of the window and view the correlation coefficient.

12. Quality Control

12.1. Quality Control specifications are summarized in Attachment I of this SOP.

12.2. Any deviations from the norm encountered while conducting this analysis must be noted in the logbook and brought to the attention of the section supervisor.

12.3. Pace Analytical Services-Florida SOP S-ALL-Q-012 Corrective and Preventative Actions or its equivalent revision or replacement outlines QA/QC items. Within the scope of SOP S-ALL-Q-012, additional guidelines are provided for data assessment, acceptance criteria for quality control measures, and contingencies for handling out-of-control data.

12.4. Pace Analytical Services-Florida SOP Pace Analytical Services-Florida SOP S-FL-Q-006 Laboratory Calculations or its equivalent revision or replacement provides guidance on statistical calculations used within the laboratory.

12.5. Samples exceeding the highest standard should be diluted and reanalyzed.

13. Method Performance

13.1. This method is validated using Method Detection Limit Studies (MDL). Guidelines for MDL studies are listed in S-ALL-Q-004 Determination of Limit of Detection and Limit of Quantitation or its equivalent revision or replacement.

13.2. Each analyst, prior to reporting analytical data for Client samples, must perform an Initial Demonstration of Capabilities (DOC) Study. Guidelines for DOC Studies are listed in SOP S-ALL-Q-020, Orientation and Training Procedures or its equivalent revision or replacement.

14. Pollution Prevention and Waste Management

14.1. Pace Analytical Services-Florida promotes pollution prevention. Whenever possible, reduce the effect of pollution by using procedures with reduced chemical exposure and volumes. Utilize the hoods in each lab to vent out fumes and perform procedures. Use judgment and good laboratory procedures while working in the laboratory. Please direct any questions to your direct supervisor. Pace Analytical Services-Florida SOP S-ALL-W-002 Waste Handling Management or its equivalent revision or replacement provides guidance on pollution prevention.

14.2. Pace Analytical Services-Florida SOP S- ALL-W-002 Waste Handling Management or its equivalent revision or replacement provides guidance on hazardous waste

15. References

15.1. USEPA, "Determination of Dissolved Hexavalent Chromium in Drinking Water, Groundwater, and Industrial Wastewater Effluents by Ion Chromatography" EPA Method 218.6 Revision 3.3

15.2. USEPA, "Determination of Hexavalent Chromium in Drinking Water by Ion Chromatography with Post-Column Derivatization and UV-Visable Spectroscopic Detection" EPA Method 218.7 Revision 1.0

15.3. "UCMR3 Laboratory Approval Requirements and Information Document" Revision 2.0, Office of Ground Water and Drinking Water US EPA, Cincinnati, OH (2012).

15.4. USEPA, "Manual for the Certification of Laboratories Analyzing Drinking Water"; Fifth Edition, January 2005.

15.5. Supplement 1 to the Fifth Edition of the Manual for the Certification of Laboratories Analyzing Drinking Water"; June 2008.

15.6. Quality Manual - Pace Analytical Services, Inc., current revision.

15.7. NELAC (TNI) Standard, most current version.

15.8. "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." 2012 Method Update Rule Federal Register, 40 CFR Part 136

15.9. Pace Analytical Services – Florida S-ALL-Q-037 Data Review Procedure, most current revision

16. Tables, Diagrams, Flowcharts, Attachments, Appendices, etc.

16.1. Attachment I: Calibration and QC Summary Table.

17. Revisions

Document Number	Reason for Change	Date
S-FL-I-050-rev.00	First issue.	16Aug2011
S-FL-I-050-rev.01	Section 2.4: Added to be consistent with other SOPs. Section 7.2: updated temperature requirement. Section 8.1: updated equipment list to match current inventory. Section 9.3: added Note. Sections 9.5 and 9.9: added word 'optional' regarding the addition of sample adjustment buffer. Sections 9.6, 9.7, 9.9, and 9.10: changed intermediate standard prep from daily to weekly. Section 9.7: changed intermediate concentration to 10ug/L. Section 9.8: revised standard concentrations to match current practice. Section 10: moved information regarding Instrument Setup, Detector Setup, AXP Setup, and Instrument Shutdown from old section 11. Sections 10.4 and 10.5: reworded Instrument Shutdown information. Section 11.2: Added section on creating a sequence. Section 11.3: Added section on creating a program. Section 11.4: Added section on creating a method. Section 11.5: Added section on running a sequence. Section 11.6: Added section on quantifying data. Section 13.2: removed specific DOC recoveries. Attachment I: changed ICAL acceptance criteria from less than 0.999 to less than 0.995. Attachment I: changed ICV and CCV acceptance criteria from 5% to 10%. Attachment I: changed LCS and MS acceptance criteria to 90-110% (from 95-105%).	28Sep2011
S-FL-I-050-rev.02	Added criteria for EPA 218.7 in support of UCMR 3	7March2012
S-FL-I-050-rev.03	Added Standard prep table to Section 9.8 Added 1mL of buffer to Section 9.12 Updated System Back pressure in Section 10.1.3 Changed units from ppt to ng/L Added Section 12 for ESI clients Updated Attachment I	28February2013
S-FL-I-050-rev.04	Section 2.3: Updated SOP Reference Section 7.1: Updated SOP Reference Scetion 9.5: Modified this section to include "Dry Solid Potassium Dichromate" Section 9.9: Modified this section to include "Dry Solid Potassium Dichromate" Section 15.2: Updated SOP reference Section 19: Added a Method Deviation Attachment 1: Calibration and QC Summary Table	21Feb2014
S-FL-I-050 rev.05	Section 7.1: Updated SOP Reference Section 7: Clarified preservation and holding time requirements Section 2.2: Added UCMR3 MRL Section 12: Added SOP references	2/27/14

S-FL-I-050 rev.06	Section 9.5: Added section for Sample Adjustment Buffer 218.6. Section 9.10: Revised section for Secondary Chromium Stock Standard. Section 11.6.8: Replaced ESI with BP. Attachment I: Revised the minimum number of standards for Initial Calibration and correlation coefficient	1/21/15
S-FL-I-050 rev.07	Section 7: Updated holding times and BPXA sample prep Section 9: Clarified standard prep and buffer options, added tables Section 8: Updated equipment list Section 10: Added clarification to instrument setting and shutdown procedures Added sections 11.2.7 – 11.2.9 Removed section 11.6.8 Revised section 12.5 Revised Table 1 blank, RLVS, RTW, and LOQ criteria	5/28/15

18. Method Deviations

Method Reference	Method Procedure	SOP Reference	Modified Procedure (Source)	Reason for Deviation
N/A	N/A	N/A	N/A	N/A

Attachment I: Calibration and QC Summary Table

QC Check	Minimum # of Standards	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	6	Initial calibration at least quarterly or as needed	$r^2 \geq 0.998$ QC must pass criteria	If it is outside of the acceptance limits, the IC must be recalibrated.
Second Source Verification (ICV)	1	Once per calibration	±10 % (218.6) Recovery, ± 15% (218.7) Recovery	If it is outside of the acceptance limits, the ICV can be re-analyzed. If it is still outside the acceptance limits the instrument must be recalibrated and the samples must be reanalyzed.
Initial, continuing & capping blank (ICB/CCB)	1	Beginning, every 10 samples & at end of analytical run	Must be <MDL.	Reanalyze the ICB/CCB. If it is still outside the acceptance limits the instrument must be recalibrated and the samples must be reanalyzed
Continuing Calibration Verification (CCV)	1	Beginning, every 10 samples & at end of analytical run (note: for 218.7, alternate level of CCV between 75 & 125)	±10 % (218.6) Recovery ± 15% (218.7) Recovery Retention Time ± 10% of calibration curve	If it is outside of the acceptance limits, the CCV can be re-analyzed. If it is still outside the acceptance limits the instrument must be recalibrated and the samples must be reanalyzed
Method Blank (MB)	1	1 per Batch of 20 samples	Must be <MDL.	If it is outside of the acceptance range limits, the MB can be reanalyzed.
Lab Control Sample (LCS)	1	1 per Batch of 20 samples	90%-110% (218.6) Recovery, 85%-115% (218.7) Recovery	Reanalyze and/or recalibrate and reanalyze the batch.
Matrix Spike Duplicate (MS/MSD)	1	2 MS/MSD sets per batch of 20 samples	For 218.6: ±10% Recovery and < 20% RPD For 218.7: ±50% recovery and ≤50% RPD for samples spiked between MRL-2xMRL ±15% Recovery and ≤15% RPD for samples spiked >2xMRL	Footnote the Matrix spike with the canned footnote "M0" which states "The spike recovery was outside acceptance limits for the MS due to matrix interference. The LCS and /or LCSD were within acceptance limits."
RLVS (reporting limit verification standard) LFB < MRL (Laboratory Fortified Blank- 218.7)	1	Daily at start of analysis	TV = 0.025ug/L +/- 50%	Verify instrument sensitivity and if acceptable, then report with qualifiers. If not, then recalibrate and reanalyze.
Method Detection Limit Studies (MDL Studies)	7 minimum, run across 3 days Suggested 9 total run over 3 days	Every 6 months, or whenever there is a significant change in background or instrument response	below RL, above 10% of the spiking level, and below the spiking level.	Identify problem & Rerun
Demonstration of Capabilities (DOC) Studies	4	Once per year for each analyst performing the analysis per matrix	Method Criteria for ICV	Identify problem & Re-run
Retention Time Window (RTW) Studies	1	Every 6 months, each time a new column type is installed, or as needed	±10% of RT of each analyte from appropriate CCV	Repeat as needed
Limit of Detection LOD	1	Once a year per matrix	TV = 1 to 3 times the MDL. Readout > 0	Identify problem, Re-prepare & Rerun
Limit of Quantitation LOQ	1	Once	TV ≥CAL1= 1 to 2 times the LOQ (same as RDL, PQL, etc.)	Identify problem, Re-prepare & Rerun If continues to fail, revise the LOQ.

Attachment C: Exhibits of Quality Control Logs and Report Forms

Exhibit 8-1. Drilling Log

SESCO group
ENVIRONMENTAL SOLUTIONS
investigation • remediation • redevelopment

SOIL BORING & MONITORING WELL CONSTRUCTION LOG

Project #: _____ Project Name: _____ Date: _____

Site Address: _____ Location ID: _____

Drilling Company: _____ Driller Name / License #: _____

Drilling Method & Equipment/Model #: _____ Length & Diameter of Coring Device (ft/in.): _____ Sample Interval (ft): _____

Total Boring Depth (ft): _____ Groundwater Encountered (ft): _____

Sample Name	Sample Media Type	Date	Time	QA/QC	Analysis

Sample Interval (ft bgs)		Core Recovery	PID Reading	Lithologic Depth (ft bgs)		Lithologic Description
From	To	(%)	(ppmv)	From	To	

Monitoring Well Construction Information

Installation Date: _____ Screened Interval (ft): _____ Well Diameter (in.): _____ Total Well Depth (ft): _____

Cover Type: _____ Sandpack Depths (ft): _____ Seal Type: _____ Seal Depths (ft): _____

SESCO Field Representative: _____ Page: _____ of _____

Exhibit 8-2. Monitoring Well Construction Log

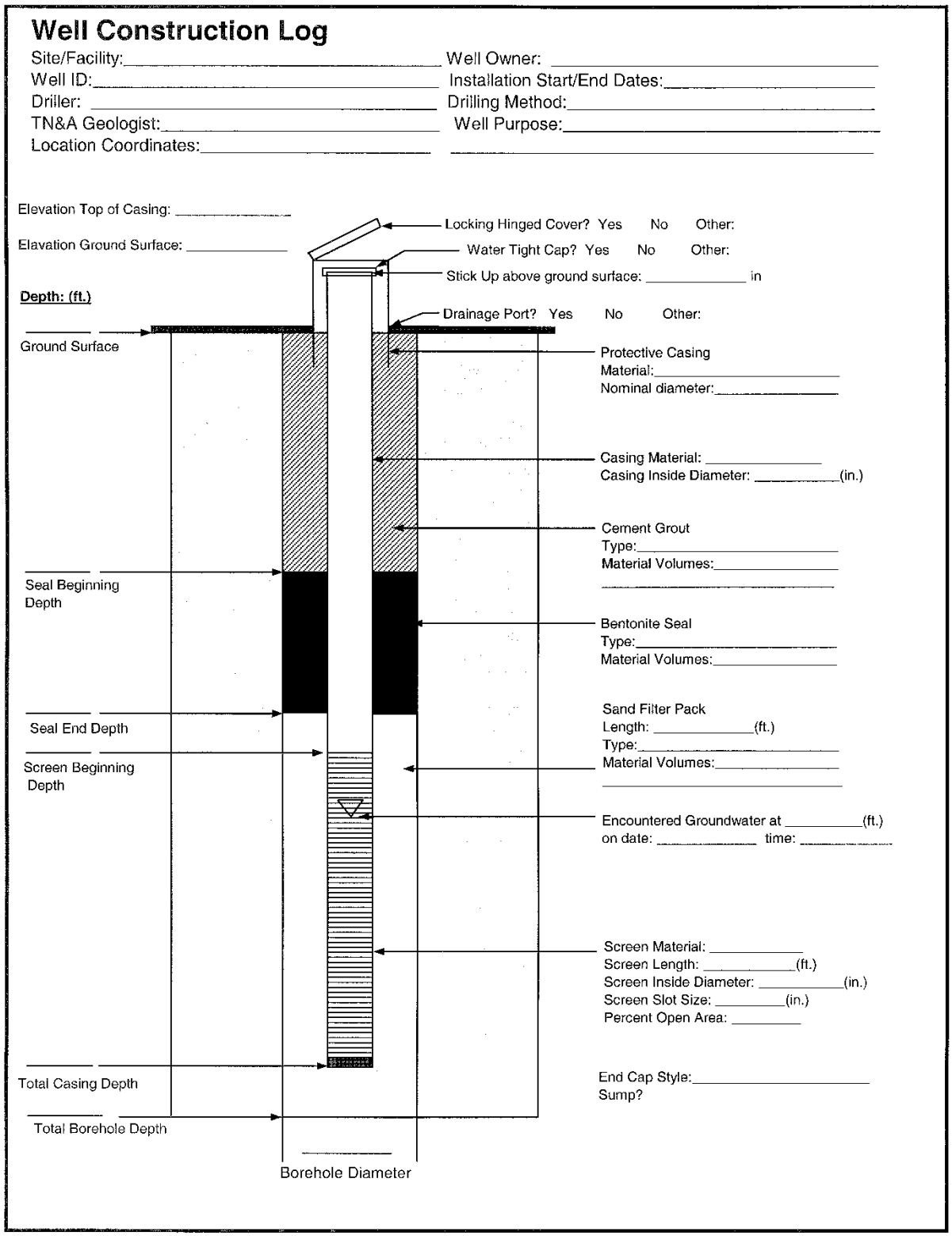
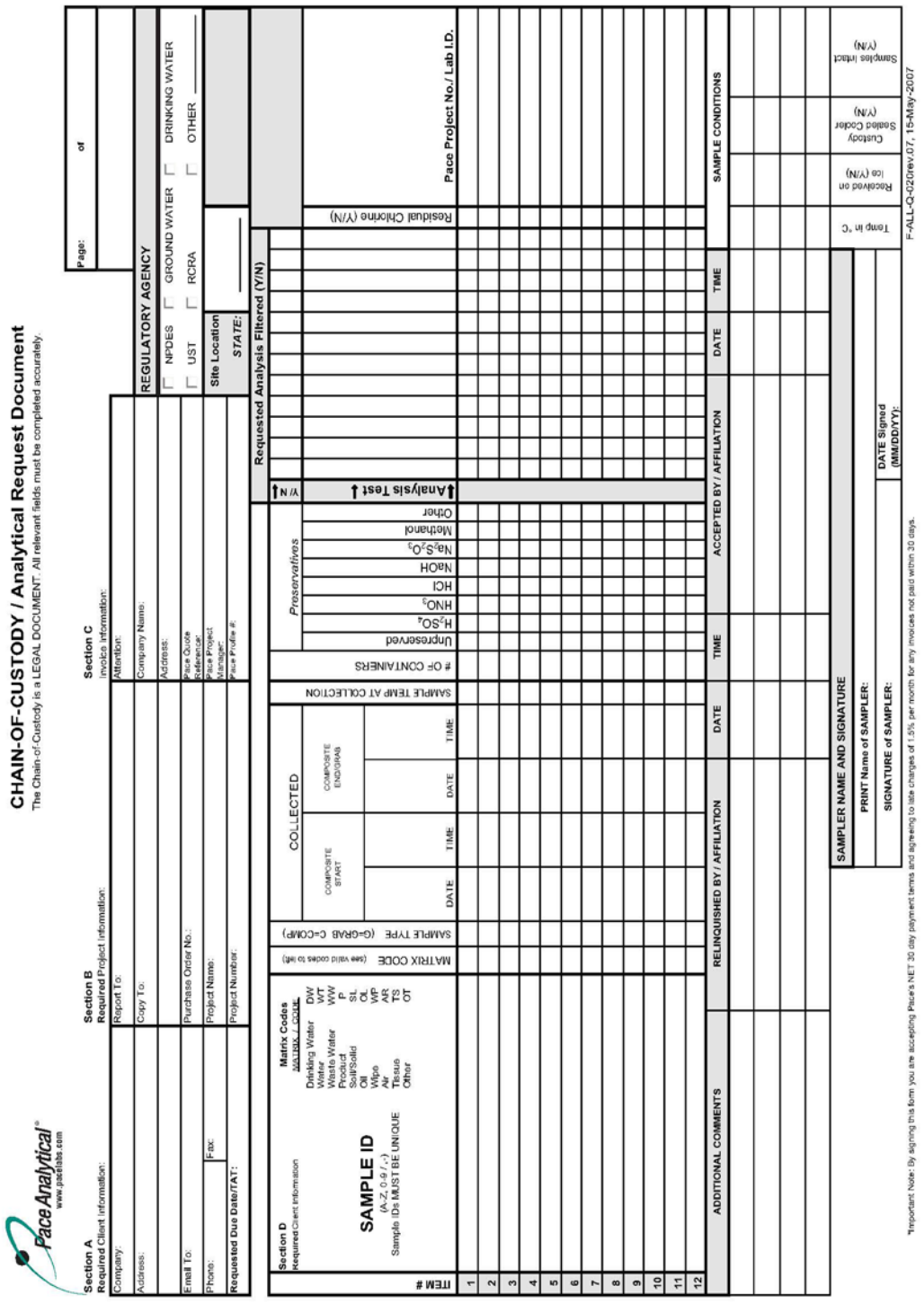


Exhibit 8-3. Example Chain of Custody Form



CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Pace Analytical® www.pacelabs.com

Section A Required Client Information: | **Section B** Required Project Information: | **Section C** Invoice Information: | Page: of

Company: | Report To: | Attention:

Address: | Copy To: | Company Name: | REGULATORY AGENCY

☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER

Email To: | Purchase Order No.: | Pace Quote Reference: | ☐ UST ☐ RCRA ☐ OTHER _____

Phone: | Fax: | Project Name: | Pace Project Manager: | Site Location STATE:

Requested Due Date/TAT: | Project Number: | Pace Profile #:

Section D Required Client Information

SAMPLE ID (A-Z, 0-9 / , -) Sample IDs MUST BE UNIQUE

Matrix Codes (MATRIX / CODE)
Drinking Water DW
Water WT
Waste Water WW
Product P
Soil/Solid SL
Oil OL
Wipe WP
Air AR
Tissue TS
Other OT

ITEM #	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test Y/N	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				
9																				
10																				
11																				
12																				

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS: Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER:

SIGNATURE of SAMPLER: | DATE Signed (MM/DD/YY):

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

F-ALL-Q-020rev.07, 15-May-2007

Exhibit 8-4. Soil/Sediment Sample Collection Log

Soil Sampling Log

CAOC ID:	
DATE:	

Sample ID	Center Point Coordinates		Depth (ft)	ASTM Soil Classification Code	Visual Description % Fragments/shot	Sieved Fraction Weight	Laboratory Analyses

*EQUIPMENT USED:*_____ **Sampler Initials:** _____

Groundwater Well Development/Purge Log

Project Name:_____ Project No._____ Phase _____ Task _____ Page ___ of ____

SITE ID: _____ **LOCATION ID:** _____ (Well Number) Target Purge Volume : _____ gal

Purging Method/Equipment: _____ Sampling Equipment/ID No: _____

Well Casing Diameter (a) in : _____ Unit Casing Volume (b): _____

Sounding (Depth to Well Bottom from TOC) (c): _____ **Static Water Level** (Depth to Water from TOC) (d): _____

Length of Static Water Column in Casing (e) = (c) - (d) = _____ - _____ = _____

Casing Water Volume (f) = (b) x (e) = _____ x _____ = _____

Casing Volumes = three x (f) _____ = _____

6" Diameter = ~1.5 gal/ft

4" Diameter = ~0.67 gal/ft

3" Diameter = ~0.37 gal/ft

2" Diameter = ~0.17 gal/ft

Date	Time 24hr	Recovery Time (min)	Purge Rate (gal/min)	Dynamic H2O Level (ft)	Volume Purged (gl)	Temp (C°)	pH	Specific Conductivity (us/cm)	Dissolved oxygen (mg/L)	Redox (mV)	Turbidity (NTU)	Water Description

Recovery Depth* (ft from TOC) : _____ Final **Recovery Time*** (min) : _____ * Taken As Final Water Level Reading and Time after sampling is complete and well has recovered.

Prepared By:_____ Checked By: _____

Exhibit 8-6. Water Level Measurement Log

Water Level Measurement Record

Date: _____ Time Begun:_____ Time End:_____

Site:_____ Project No: _____

Well ID	Elevation Top of Casing (ft)	Depth to Water (ft)	Water Elevation (ft)	Depth to Well Bottom (ft)	Well Integrity			
					Properly capped & locked?	Surface seal intact?	Frost heave or tampering?	Obstruction in well?

Additional Comments: _____

Prepared by: _____ Date: _____

Exhibit 8-7. Inspection Log

MAINTENANCE AND CALIBRATION RECORD

DATE: TIME:	
EMPLOYEE NAME:	EQUIPMENT TITLE:
CONTRACT/PROJECT:	EQUIPMENT SERIAL #
ACTIVITY:	EQUIPMENT ID #:
MAINTENANCE PERFORMED:	COMMENTS:
SIGNATURE:	DATE:

CALIBRATION/FIELD CHECK

CALIBRATION STANDARD:	CONCENTRATION OF STANDARD:
LOT NUMBER OF CALIBRATION:	EXPIRATION DATE OF CALIBRATION STANDARD:
PRE-CALIBRATION READING:	POST-CALIBRATION READING:
PRE-FIELD CHECK READING:	POST-FIELD CHECK READING:
ADJUSTMENT (S):	
CALIBRATION: ☐ PASSED ☐ FAILED	
COMMENTS:	
SIGNATURE:	DATE:

Exhibit 8-8. Field Test Results Log

EXAMPLE - FIELD TEST FORM

Landfill Gas Monitoring Record

Page of

Date: _____
Circle: Sun Mon Tues Wed Thur Fri Sat

Site: _____ Project No: _____

Sampler: _____

Air Temp: _____ % Humidity: _____

Barometric Pressure: _____ Source of Data: _____

Approx. Wind Speed: _____

				Measurements					
Gas Vent ID	Time	Comments on Vent Well Intergrity	Screen Depth (ft bgs)	PID Reading	Oxygen	CO_2	Temp C	CH_4 specify % Vol. or % LEL	Pressure Reading (mm Hg)

Equipment used: _____

Signature: _____ Date: _____

Exhibit 8-9 QC Inspection Report

QC Completion Inspection Date _____

USACE COR:	Inspectors:
Project:	
Project Number:	
Contract Number:	

ITEM INSPECTED	DEFICIENCY	ANTICIPATED CORRECTION DATE

Exhibit 8-10. Variance Form

VARIANCE FORM

VARIANCE No. (XXXX) _____

PROJECT NUMBER _____ PAGE ___ OF ___

PROJECT NAME _____ DATE _____

VARIANCE (INCLUDE JUSTIFICATION)

APPLICABLE DOCUMENT:

cc:

REQUESTED BY _____ DATE _____

APPROVED BY _____ DATE _____

Project Manager

_____ DATE _____

QA/QC Officer

_____ DATE _____

Exhibit 8-11. Variance Log

VARIANCE LOG
CHRONOLOGIC LIST OF PROJECT VARIANCES

PROJECT MANAGER _____ PAGE ____ OF ____

PROJECT NAME _____

DATE	VARIANCE GRANTED AND APPLICABLE DOCUMENT	RESPONSIBLE INDIVIDUAL

Exhibit 8-12. Nonconformance Report

NONCONFORMANCE REPORT

NR NO. _____

PROJECT NUMBER _____ PAGE ___ OF ___

PROJECT NAME _____ DATE: _____

1. NONCONFORMANCE DESCRIPTION

IDENTIFIED BY: _____ DATE: _____

2. PROPOSED CORRECTIVE ACTION, INCLUDING INITIATION AND COMPLETION DATES

TO BE PERFORMED BY: _____

3. APPROVAL FOR PROPOSED CORRECTIVE ACTION

Project Manager: _____ DATE: _____

QA/QC Officer: _____ DATE: _____

4. CORRECTIVE ACTION TAKEN (IF DIFFERENT FROM THAT PROPOSED)

5. CORRECTIVE ACTION COMPLETE

PERFORMED BY: _____ DATE: _____

VERIFIED BY: _____ DATE: _____

cc:

PROGRAM MANAGER:
PROJECT MANAGER:
QA/QC OFFICER:
OTHER:

Exhibit 8-13. Daily Quality Control Report

CONTRACTOR QUALITY CONTROL REPORT (ATTACH ADDITIONAL SHEETS IF NECESSARY)		DATE
CONTRACT NO.	TITLE AND LOCATION	REPORT NO.

CONTRACTOR Oneida Total Integrated Enterprises	FIELD SUPERVISOR/PROJECT MANAGER
AM WEATHER sun PM WEATHER	MAX TEMP F MIN TEMP F

WORK PERFORMED TODAY

WORK LOCATION AND DESCRIPTION	EMPLOYER	NUMBER	TRADE	HRS

WAS A JOB SAFETY MEETING HELD THIS DATE? ☐ YES ☐ NO (If YES attach a copy of the meeting minutes)	Total Work Hours on Job Site This Date	
Were there any lost time accidents? ☐ YES x☐ NO (If YES attach a copy of the OSHA report)	Cumulative Total of Work Hours From Previous Report	
Was hazardous material/waste released into the Environment ☐ YES x☐ NO (If YES attach description of incident and proposed action)	Total Work Hours From Start of Construction	

WAS TRENCHING/SCAVVOD/HV ELECTRIC/HIGH WORK DONE? ☐ YES ☐ NO
(If YES attach statement or checklist showing inspection performed)

List safety actions taken today/safety inspection conducted ☐ Safety requirements have been met.

Equipment/material received today to be incorporated in job

Work performed today

Equipment on site today, include number of hours used today

Verbal instructions received: (List any instruction given by the Government personnel on construction deficiencies, retesting required, etc., with action to be taken.)

Test required by plans and/or specifications performed and results of tests
List the total number samples collected and tested for the day: COLLECTED: TESTED: AMPLIFYING INFO:
List the total amount of waste(s) removed from the site: LIQUID (bb/gal): SOLIDS (yds/tons):
List the following transportation and/or disposal information to date for the project

QUALITY	ID NO.	MATERIAL	MANIFEST NO.	DISPOSAL LOCATION

List all safety violations observed and corrective actions taken:
Submittal Action:
Remarks: _____ _____ CONTRACTOR/SUPERVISOR DATE